Atomic Absorption Spectrophotometer

AA990

Operation Instruction Manual

Precautions for Safe Operation and Warning Marking

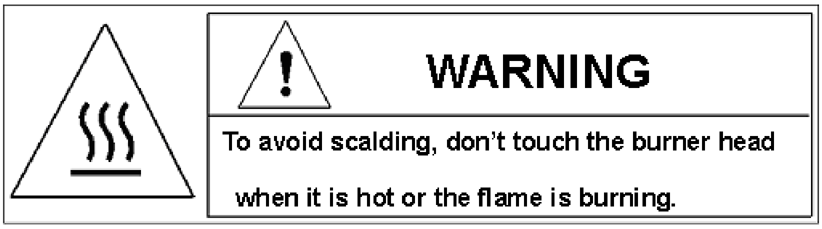
The flame system of the instrument uses combustible and detonatable high pure acetylene gas and 220V power supply. Please read the instruction manual and warning labels carefully before operating the instrument. And please strictly observe the following precautions and pay attention to the safety prompting messages of the instrument.

Daily operation cautions:

(1) Exhaust duct should be installed above the burner to exhaust the combustion gas;
(2) Do not smoke or use open fire near the instrument;
(3) Extinguish the flame before living the gauge house;
(4) The fluoridebearing samples cannot be measured when using glass nebulizer.

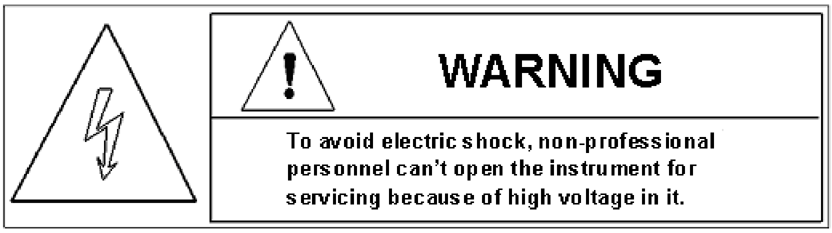
Warning labels:

- **This label is on the decorative board front of the instrument.**



To avoid scalding, don’t touch the burner head when it is hot or the flame is burning.

- **This label is on the left bottom rear of the instrument.**



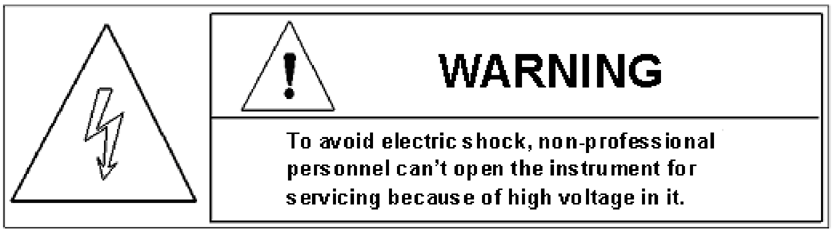
To avoid electric shock, non-professional personnel can’t open the instrument for servicing because of high voltage in it.

- **This label is on the right bottom rear of the instrument.**

WARNING

Verify that there are no leaks in any of the gas lines, igniting is strictly prohibited if there are acetylene leaks, otherwise it will explode.

- **This label is on the power supply of the graphite furnace**



WARNING

To avoid electric shock, non-professional personnel can't open the instrument for servicing because of high voltage in it.

- **This label is hung on the acetylene cylinder.**

WARNING

Verify that there is no leak before opening the acetylene cylinder, igniting is strictly prohibited if there is acetylene leak, otherwise it will explode.

Precautions on Handling High Pressure Gas

The flame system of the instrument uses combustible and detonatable high pure acetylene gas, so cautionary items in this manual should be read carefully.

(1) Install the gas cylinders in the airy outdoors where they are not exposed to direct sunlight. Then pipe it to the gauge house. Do not place the acetylene gas cylinder and instrument in the same place.

(2) Take care that gas cylinder become to no hotter than 40^0C. And do not allow any flame within 3 meters of the gas cylinder.

(3) Fasten gas cylinder with tightwire so that they cannot drop or fall over. Always keep the gas cylinders in a vertical position.

(4) Use dedicated voltage regulator for the acetylene gas. Do not use pipes made from copper, silver, mercury, or their alloys in order to prevent the formation of metal-acetylides. Decompositon of such compounds could result in a “decompositon explosion”resulting from a shock.

(5) After the gas was used, be sure to close the main valve as well as the stop valve.

(6) Periodically inspect the pressure gauge to maintain its normal function.

(7) Before install the voltage regulator, wipe the dust at the outlet of the gas cylinder.

(8) Before opening the cylinder, be sure to check that the stop valve is closed. Turn the secondary pressure-adjusting valve fully to the left, and open the cylinder using a special handle. Even if the main valve is too tight to open, do not hit the handle or main valve with the hammer or spanner. Then, apply soapy water to the connection of the pressure regulator to check if no gas leak is generated.

(9) Be sure to rotate the main valve of the cylinder for acetylene gas by 1 –2 rotations to avoid the outflow of acetone from inside of the cylinder.

(10) Adjust the secondary pressure-adjusting valve clockwise to 0.07Mpa. Do not make it over 0.1Mpa. Acetylene gas is not stable at high-pressure status. When the pressure is over 0.1Mpa, it may be result in a “decompositon explosion” if it heated, impacted, or rubbed even if without oxygen and air. The “dissolving acetylene” is rather stable, but you should be careful to the high voltage secondary pressure-adjusting valve when the pipe is to a certain length.

(11) The acetylene gas cylinder contains solvents such as acetone. If the primary pressure reduced below 0.5Mpa, it should be replaced with a new cylinder in order to prevent the outflow of solvents.

Security Mechanism and Operation Precautions

The instrument has complete security mechanism, but the user also should strictly observe the security operation standard. The security mechanism should always be checked. The security mechanism and the precautions are shown as follows:

1. Gas leaking alertor:

The gas leaking alertor, made of the combustible gas sensor, is near the gas inlet in the instrument. It begin to work when switch on the external power (no matter whether the instrument power is on or not). It provides audible alarm in addition to alarm safety chain protection.

Warning: whenever it alarms, please turn off the acetylene first, find out the reasons and solve the problems thoroughly, then turn on the instrument again.

Daily care: you must check the acetylene alarm system within a month; the method is as follows:

- Facing the rear cover of the instrument, disassemble the lower leftmost bolt, which is used to fix the cover of the instrument in the vertical orientation.
- Electrify the instrument. Two minutes later, take out saturation alcohol gas *****5ml.
- Insert the injector into the hole of the disassembling bolt, then inject.
- The audible alarm will start to work in two minutes.

***saturation alcohol gas： Refers to the gas in the alcohol bottle. (The alcohol concentration is more than 99%)**

2. Air pressure monitoring:

When the provided air pressure is lower than 0.2Mpa， the instrument will alarm.

3. Burner head position monitoring:

Check whether the burner head is installed in the right place, or the instrument will not be lit.

4. Waste liquid level monitoring:

Check whether the waste liquid level is enough.

5. Flame monitoring:

If the flame extinguished abnormally, the instrument will give an alarm.

6. Extinguishing the flame in emergency:

If the abnormal situation emerged, press the button of extinguishing flame in emergency, and turn off the acetylene valve as soon as possible.

Points for attention:

1. The safety system of flame atomizer is effective when starting ignition and in flame working mode. When all the safety mechanisms are in the normal conditions, it can begin to work;
2. The flame will be extinguished safely as long as there are any abnormality existing in safety system, and the display screen will provide automatic alarm signal;
3. If the emergency situation or abnormal situation emerged, please handle it referring to the emergency action.
4. Check the instrument carefully before reuse it. Contact with service personnel if necessary.

Emergency action:

1. Turn off the power switch of the atomic absorption spectrophotometer.
2. Turn off the main valve of the gas supply piping for acetylene, nitrous oxide, hydrogen, air and argon.
3. Turn off the main valve of the circulating cooling pipe.

General precautions

In order to use the instrument safely, be sure to observe the following cautions. If not observed, the safety of the equipment may be compromised.

(1) When pressing the ignition button (at igniting), never put your face or hands in the burner compartment. Do not look into from above the combustion chamber and do not hold the hands over the combustion chamber. When the nitrous oxide – acetylene gas flame is ignited, the flame goes up to the level of approx.40cm from the top of the equipment. Care should be taken. Before igniting, be sure to close the flame shield (combustion chamber door).

(2) Do not ignite a flame unless the burner is mounted to the chamber.
(3) Do not ignite a flame unless the nebulizer is mounted to the chamber.
(4) Do not ignite a flame unless water is filled in the drain tank.
(5) Do not remove the nebulizer from the chamber during combustion.
(6) Do not remove the drain tube form the chamber during combustion.
(7) Do not remove the burner during combustion.
(8) Do not touch the burner while it is hot.
(9) Do not touch the deuterium lamp while it is hot.
(10) Do not use the regular burner to ignite the acetylene-nitrogen oxide flame.
(11) Do not put fingers or hands in the hole of lamp turret, igniter, and flame monitor.
(12) Do not put any foreign objects into the hole of lamp turret.
(13) Do not touch the end of cleaning wire for nebulizer capillary directly by hand. Wire may stick your finger.
(14) Do not remove the thermal shield plate when using AA-990. Heat of flame is introduced to the inside and the measurement may be unreliable.
(15) Check the setting of the gas flow rate before igniting a flame.
(16) Do not touch the flame shield during combustion.
(17) Do not touch the flame shield and burner directly by hand in about 20 minutes after extinguishments of the flame.
(18) Do not hold any substance over the flame.
(19) Do not use the flame but for analysis.
(20) Do not connect the power cable to the outlet that is not provided with grounding terminals.
(21) Do not throw away the hollow cathode lams randomly. Electrodes of some hollow cathode lamps contain harmful metal elements and some may ignite when touched with air or water.

Contents

Chapter 1 Overview

Preface

The AA-990 Atomic Absorption Spectrophotometer is a full automation intelligence-type instrument for flame and/or graphite furnace analysis developed by our company. It is a user-friendly, easy-to-use instrument controlled by PC with MS windows as the operation system. The AA-990 Atomic Absorption Spectrophotometer incorporates two background correction functions, the deuterium lamp method and the self-reversal method. By use of advanced electronic circuits and RS-232 serial communication port control, the unit allows you to make an automatic setting or adjustment for wavelength scan, peak-search, slit width switch-over, element lamp turret switch back and forth, height/position parameter setting and adjustment of the atomizer, the fuel and supporting gas flow rate, lamp current and as well as the negative voltage of the photomultiplier etc. It is also provides referential operating parameters in different measurement modes for all the analyzed elements. Users may modify these parameters according to their individual requirements for analysis, save them together with the calibration curve and the measurement results, and recall them for use when needed. The instrument switches flame/graphite furnace atomizers automatically, and also supports the flame/graphite furnace auto-samplers.

AA-990 atomic absorption spectrophotometer provides configuration options of flame and graphite furnace analysis at your own discretion. Manual and automatic switching of the atomizer are also provided. There are four kinds of configuration:

1. Flame；
2. Graphite furnace；
3. Manual switching of flame / graphite furnace；
4. Automatic switching of flame / graphite furnace；

Users can choose flame auto-sampler and graphite furnace auto-sampler according to their instrument.

Note: the user can operate the specific configured instrument by consulting the corresponding paragraphs and chapters of the instruction manual.

1.1 Scope of applications

Atomic absorption spectrophotometer is used for the analysis of minim inorganic elements to trace inorganic elements. It can perform qualitative analysis and quantitative determination. It is widely used in environment protection, medical care, metallurgy, geology, foods, petrochemical industry, agriculture, industry and many other applications.

1.2 Basic working principle

The light source of hollow cathode lamp radiates the characteristic spectrum of the target element. Then the measured element absorbs characteristic spectrum after atomization. The content of the measured element can be calculated by measuring the absorbance of the spectrum.

1.3 Features

1. Function：The AA-990 series Atomic Absorption Spectrophotometer allows you to make atomic absorption analysis by Flame and/or Graphite Furnace method depending on the configuration of users' choice.
2. The AA-990 series Atomic Absorption Spectrophotometer incorporates two background correction function (according to the configuration of user's choice), the D2 method (Deuterium Lamp method) and/or the SR method (Self-Reversal method), endowing them with strong background correction capability and better flexibility or adaptability, which provides users with selection of the appropriate method for the measurement sample.
3. The AA-990 series Atomic Absorption Spectrophotometer integrates a general-purpose PC for the full automatic control over the main unit of the Atomic Absorption Spectrophotometer, which runs a special AAWin software program with Microsoft windows as the operating system, providing users with a variety of strong data processing and analysis functions including transformation of measurement results and all kinds of analytical curves for displaying, printing, saving/storing and loading for re-use. The software also provides a specialist system, which offers users referential conditions of analysis for each element. In addition, the computer may run separately for general-use when operated offline.
4. The AA-990 series Atomic Absorption Spectrophotometer provides users with a series of fully automated functions for the instrumental control, allowing customers to realize all sorts of parameter setting and motor drive control through the keyboard operation. They are as follows:
   （1）Automatic setting the fuel gas flow (C2H2) and optimizing the gas ratio of the combustion to the supporting；
   （2）Automatic setting and adjusting the burner height for selecting the optimal analytical conditions；
   （3）Automatic loading of the element lamp through an eight-hole multiple turret holder and simultaneously lighting two lamps with one for warm-up；
   （4）Automatic switchover of the atomizer between the two analytical methods: flame and graphite furnace, when the AA-990 Flame/Graphite Furnace dual-mode Atomic

Absorption Spectrophotometer is used；

（5）Automatic switching the half-transmitting-half-reflecting mirror allowing the light beam of deuterium lamp entering into optical path when D2 lamp background correction mode is used；

（6）Automatic wavelength scanning and peak-searching;

（7）Automatic switchover of bandwidth with five-level parameter setting；

（8）Automatic setting the negative voltage for photomultiplier and the lamp current, automatic beam-balance, automatic ignition and interlock protection for abnormal extinguish etc.；

（9）Enabling fine adjustment for a variety of instrumental parameters etc.

1.4 Specifications

Item		**Specification**
Wavelength range		190 - 900nm；
Light source	Type	Hollow cathode lamp (HCL), D_2 lamp；
	Modulation mode	Square wave pulse；
	Modulation frequency	100Hz (Self-Reversal background correction mode)，400Hz (D_2 lamp background correction mode)；
Optical System	Model	Czerny-Turner monochromator
	Chromatic/optical dispersing component	Planar diffraction grating；
	Grating	1800 grooves/mm；
	Blazed wavelength	250nm；
	Focus	300mm；
	Bandwidth	0.1、0.2、0.4、1.0、2.0nm；
	Scan mode	Automatic；
Photometer type		Single beam；
Atomization system		Titanium metal burner head(single slit: 100mm x 0.6mm)；rot-proof spray chamber，high efficiency glass nebulizer；burner height auto-adjustable；
Data Processing System	Measurement mode	Absorbance，concentration，transmittance，emission intensity；
	Read-out mode	Continuous，peak height, peak area；
	Output mode	On-line instrument operating status Display, CRT Display & Printout of measurement results, calibration curve, signal profile, instrumental parameters and all other kinds of graph etc.;

	Data processing functions	Multi-standard calibration method, standard addition method, interpolation method； Integration time (0.1~20 seconds)； Sampling delay (0~20 seconds)，number of standard samples (1~8)，number of samples (0~100)；slope，mean value，standard deviation (S.D.)，relative standard deviation (R.S.D.) etc., correlation coefficient and concentration values；
	Data storage	Analytical results, instrumental and measurement parameters, signal profile and calibration curves all can be stored in the computer hard disk；
Power requirements		Power supply: Main unit 220Ｖ，50Hz，200W； graphite furnace power supply 220V，the maximum instantaneous power 5KW；
Instrument dimensions		110cm×50cm×45cm，weight：75Kg；

1.5 Technical descriptions

1. Wavelength range and its precision：

（1）wavelength range：190-900nm；

（2）device：C-T type of achromatic aberration

（3）wavelength accuracy：±0.25nm；

（4）resolution：better than 0.3nm；

（5）bandwidth：0.1, 0.2, 0.4, 1.0 and 2.0nm (with five-level automatic switch-over)；

2. Instrument stability：baseline drift less than ±0.005A in 30 minutes

1.6 Standard configuration

1. Main unit: 1 set

2. Special software：AAWin 1 set

3.Standard accessories：

(1) Hollow cathode lamp for Hg, Cu, Mn, Cd (Cd lamp with graphite furnace) 1 for each

(2) Low noise oil free air compressor 1 unit

(3) Spare nebulizer 1 piece

4.Some of the optional configuration:

- Flame Auto-sampler；
- Graphite Furnace Auto-sampler；
- Automatic Temperature Controlled Circulating Cooling Water System；

Notice：The standard configuration is subject to change without notice. Please check the configuration according to your packing list. You can contact with the manufacture to confirm your optional configuration at any time.

Chapter 2 Installation and acceptance

2.1 Laboratory requirements

2.1.1. Laboratory environment conditions:

The installation environment should be proper. The location should be kept away from a strong electromagnetic field, a strong calorific radiation or strong light. The laboratory should also not be situated near to the equipment or workshop that generate strong vibrations. It should also be kept from the influence from direct sunshine, smoke dust, dirty air current and vapor. The AAS laboratory should be separated from the chemical treatment room and the emission spectrometric laboratory so as to guard against acid erosion or interference from a strong magnetic field. The internal environment of the laboratory should also always be kept clean with appropriate temperature (at 15-30°C) and humidity (below 70% relative humidity).

2.1.2 Equipment requirements for the laboratory

The dimensions of the main unit are 1100mm X 500mm X 450mm. And the dimensions of the graphite furnace power supply are 700 X 500mm X 450mm. The laboratory bench should be big enough to install the instrument and computer etc. It (cement bench is the best choice), recommended 0.75 meters height, also should be stable and solid with a flat face. Around the bench, a space at least larger than half a meter should be left to facilitate the operation and maintenance.

Exhaust equipment should be installed in the laboratory (see figure 2-1) with the volume of air discharged enough to catch up a piece of newspaper slightly larger than the suction vent and attached below the opening.

2.1.3 Laboratory Power Distribution Requirements

Power incoming line should adopt the three-phase-four-line of AC 50HZ, with a safe earth wire. The instrument and its accessory instruments uses 220V power supply by standard plugs, so the switchboard is suggested to use. See figure 2.

The wiring requirements: the main unit of the AAS should be used with air compressor and power supply of graphite furnace phase splittingly.

* The main unit of AAS uses one phase, 220V±10%/5A, with power desirably supplied through a purified voltage regulator with its power above 1.5KW.

* Air compressor etc. accessory equipment uses one phase, 220V/10A.

* The power supply of graphite furnace should use another one phase, more than 220V/40A.

In order to ensure reliable connection, the power supply of graphite furnace should use 380V/25A with four holes power plug to lead out (See figure 2-2).

2.1.4 Gas source requirements

* Oil free air compressor: suggested to buy with the instrument.

* Acetylene gas: the purity should be no less than 99.9%， and the acetylene cylinder should use special acetylene reducing valve. A special gas cylinder storage room is needed, but the gas on-off valve in the laboratory is also need.

* Argon gas: when configured with graphite furnace system, the argon gas with 99.99% purity should be prepared as a protection gas. And a special gas reducing-valve is also needed.

2.1.5 Water source requirements

When configured with graphite furnace system, the on-off running water is needed, such as tap water, and the flow rate is more than 1.5L/min. When configured with graphite furnace auto-sampler, the automatic temperature controlled circulating cooling water system is recommended.

2.1.6 Solution requirements

DI water, copper standard solution, cadmium standard solution (used for graphite furnace); Necessary measuring tools and containers for the analysis laboratory

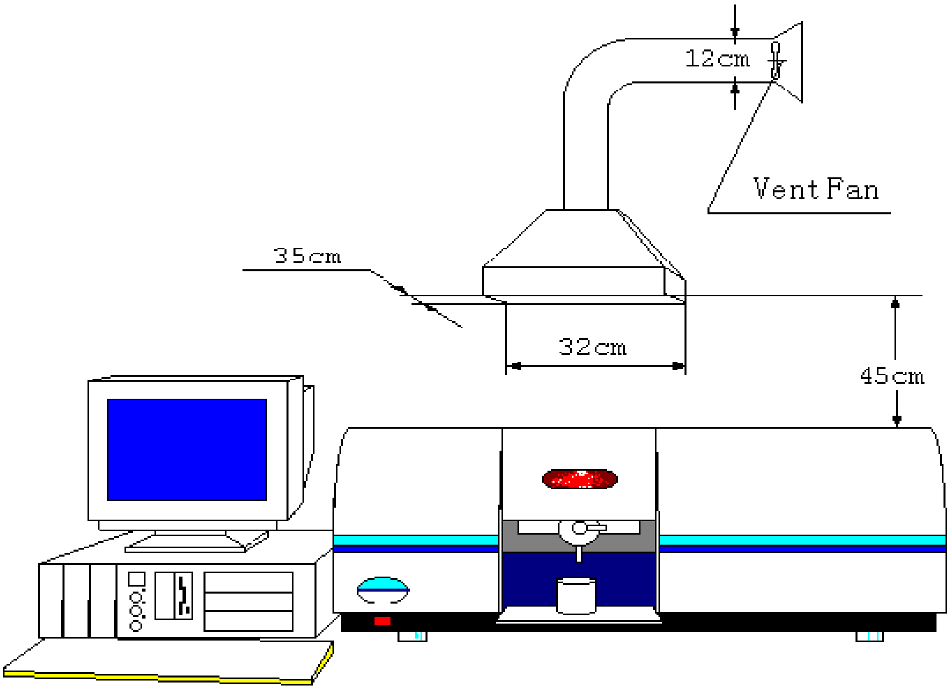


Fig. 2-1 The scheme of installation of ventilation equipment

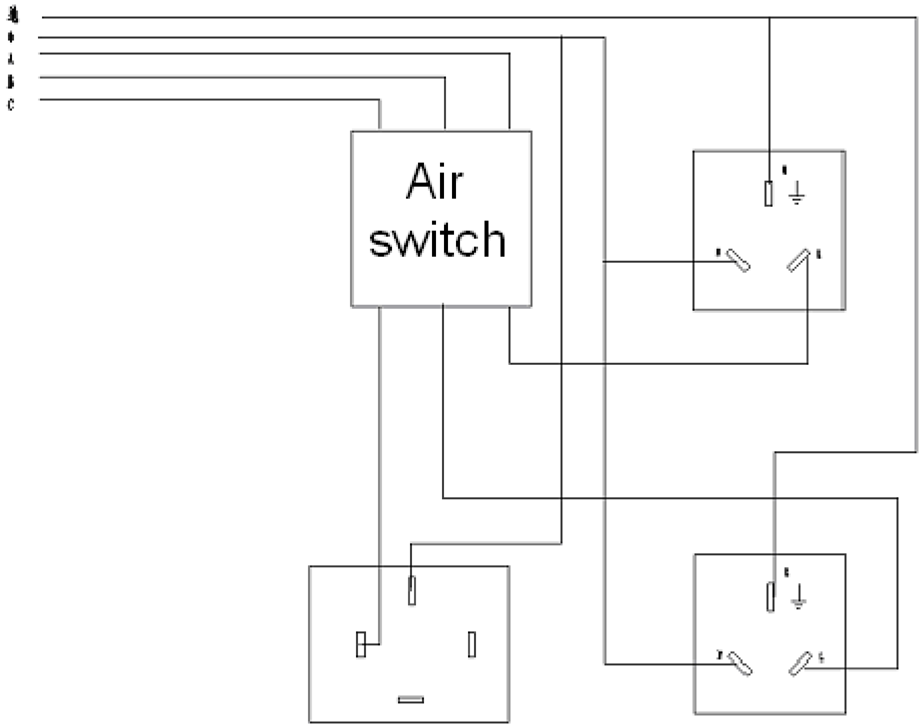


Fig. 2-2 The connection scheme of switchboard

- **Note: choose the appropriate air switch or the equivalent switch and protection equipment.**
- **The switchboard should be designed and installed by professional electrician.**

2.2 Preparations before installation

1.Unpacking for examination and acceptance. After unpacking the instrument, please check item by item against the packing list to see whether the main unit, accessories, parts, spare parts and the operation manual are complete or not. Meanwhile, please check whether any damage is done to the appearance of the instrument. While finding out any problem, please contact the supplier promptly to ascertain the reason.

2. Please read carefully the operation manual to familiarize you with operating principles and structure of the instrument and learn its use and installation steps.

3. Please get ready for the power supply, the fuel gas tubing, the waste fluid pail and the element lamps according to the laboratory requirements in 2.1.

4. Please notify the supplier in advance for a technician to be sent over for instrument installation and testing.

2.3 Installation

2.3.1 Connection

This chapter is for installation engineers., and the user can read it for reference.

1. Connecting the computer system:

The computer system is composed of a main frame, a display, a keyboard and a printer. Connect the keyboard, the display and the printer to the respective interfaces on the computer mainframe. Then connect the printer power cord at the back of the printer to the 220V power supply source. One end of the power cord for computer is to be connected to the power supply socket at the back of the computer mainframe with the other end connected to the 220V power supply source.

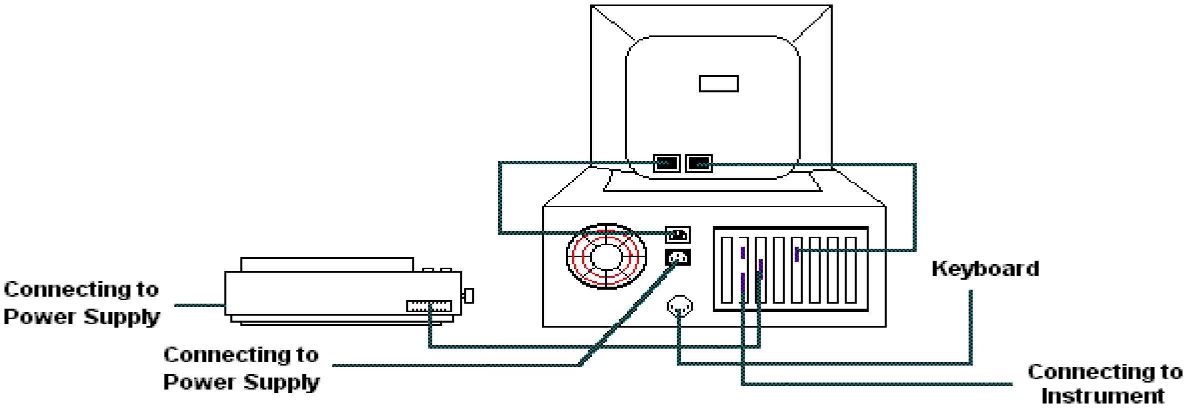


Figure 2-3 Sketch Map for Computer Connection

2. Installing the atomic absorption main unit:

Put the main unit on the laboratory bench carefully for connecting the power supply and gas piping, as shown in Fig. 2-4.

(1) Connection of the main unit with the power supply: At the lower right side of the back plate of the main unit can be found a socket (See Figure 2-4). Connect one end of the supplied power cord to this socket and its other end to the socket of the power supply.

(2) Connection of gas piping

(a) Connection of air piping: Connect one end of a black air tube with appropriate diameter and length to the outlet of air compressor (equipped with a special gas-piping interface for your ready insertion). And insert its other end into the inlet for air compressor on the back plate of main unit (See Figure 2-4).

(b) Connection of acetylene gas piping: Connect one end of the yellow/red acetylene gas tube into the outlet of the steel acetylene cylinder (equipped with a special gas-piping interface for your ready insertion). And insert its other end into the inlet for the acetylene gas on the back plate of atomic absorption main unit.

(c) Connection of waste fluid piping:

- Fasten the liquid-level monitoring device;
- Install and fasten the transparent tube at the bottom of the liquid-level monitoring device by using hose clips.
- Fasten one end of the transparent tube to the upper joint of the liquid-level monitoring device by hose clips and the other end of the tube to the waste liquid container.
- Top up clean water to the liquid-level monitoring device.

Note: the liquid-level monitoring device must be filled with water. Please check the liquid level when use flame analysis each time. (It can’t be ignited or will cause abnormal extinguish if the liquid level is too low.)

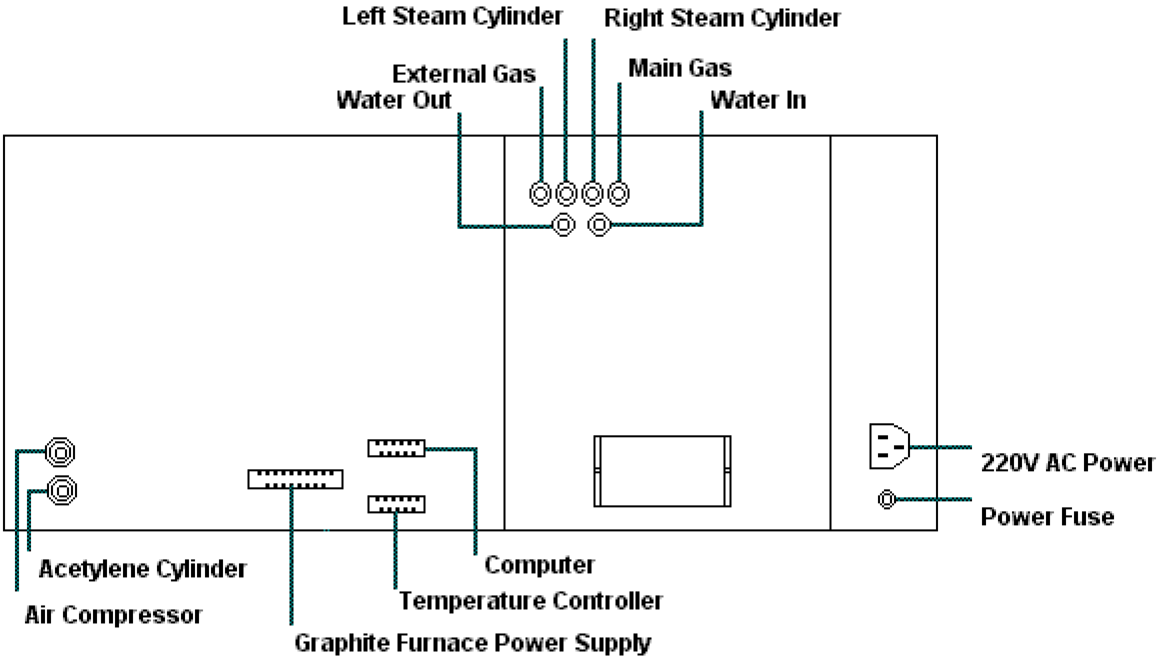


Fig. 2-4 The rear side of the main unit of AAS

3. Installation of the graphite furnace main unit system

(1) Connection of the main unit with the graphite furnace power supply (See figure 2-4, 2-5).

(2) Connection for the protection gas piping (See figure 2-4, 2-5).

(3) Connection for the cooling water piping (See figure 2-4, 2-5).

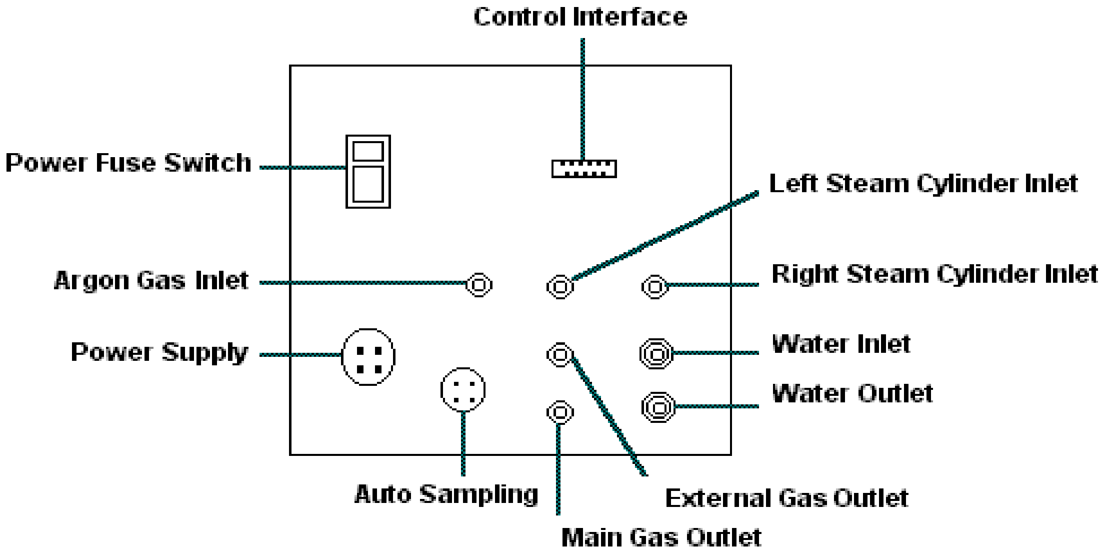


Figure 2-5 Sketch Map of Graphite Furnace power Connection

4. Other connection:

Connect the communication cord to the control signal cord of graphite furnace. Check the power supply system, and then connect the various power supply cords if the power supply system is qualified.

5. Installing hollow cathode lamps:

Find the eight-lamp turret holder in the light source chamber at the upper left side of the system. Select the corresponding element lamps according to the elements to be measured and put these lamps onto the turret before taking measurements. You may insert up to eight lamps at a time. Just insert lamps one by one into the lamp sockets on the turret, then plug these sockets with the numbers of 1-8 in sequence into the holes of the lamp turret holder with not too deep insertion.

2.3.2 Software installation

2.3.2.1 System configuration

Windows98/2000/XP are all acceptable for AAWin.

- **Base configuration:**

IBM-PC compatible computer, 400MHZ processor, 32M memory, and 10G hard disk

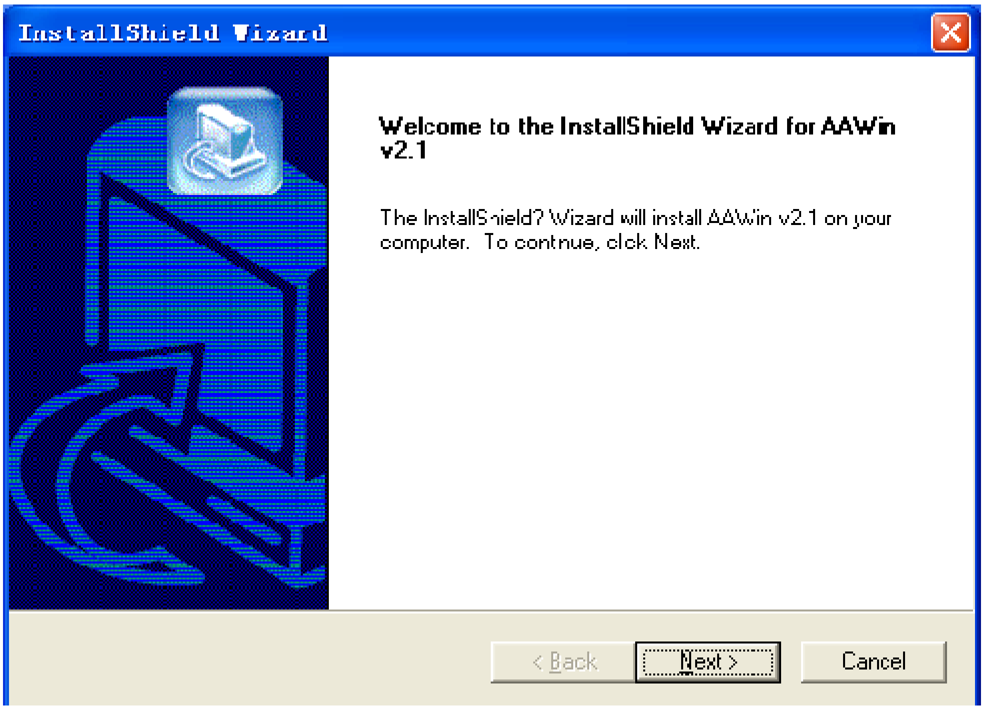
- **Recommended configuration:**

IBM-PC compatible computer, over 800MHZ processor, over 64M memory, and 10G hard disk

2.3.2.2 Installation

There are three pieces of floppy disks (or one piece of CD) for installation. Please insert

AAWin installation disk No.1 into your CD-ROM. Press on “start” to “run” ordinal, and then input “X: Setup” in the current interface. Finally, hit the button “ confirm” to install the program. There is another way for doing that by opening the driver in the source controller and hit “Setup.Exe” twice.



Welcome to the InstallShield Wizard for AAWin v2.1

The InstallShield? Wizard will install AAWin v2.1 on your computer. To continue, click Next.

Figure 2-6 Installation

After the program runs, you can see the interface as fig. 2-6. You can do as the system hints and you can find the fold of “AAWin for AA-990” in the “program” menu, the AAWin software can be run as long as you hit “AAWin” in which the fold. It also can run by clicking the shortcut on the desktop. If you want to delete “AAWin” software from your system, you can just delete “AAWin” software from the “add &delete” in the control board.

2.3.2.3 Document system

1. The AAWin system includes the following:
   - AMD file: A file for data measuring. All the metrical data and the calibration curve are kept in this file.
   - SGL file: A file of the signal curve. The data of signal curve is kept in this file.
2. In addition to the custom files there are some files that had been defined already. Stated as

follows:

- .DOC file: Output Microsoft Word documents with [Export Files].
- .XLS file: Output Microsoft Excel worksheets with [Export Files].
- .TXT file: Output Text files with [Export Files].

2.4 Acceptance

After installation, the instrument can be adjusted and accepted. But before acceptance, the operator must read the instruction manual carefully to get familiar with the operation and acceptance method. The user should check the instrument item by item according to the installation acceptance certificate, which also should be filled.

The following provides the testing methods of some specifications:

1) Wavelength accuracy and reproducibility

1.1 Testing tools

Hallow cathode lamp: Mercury (Hg)

1.2 Testing method

Start with wavelength correction by using Hg at first, then choose 0.2 nm as the bandwidth to search the peaks at 253.7 nm，546.1 nm，871.6 nm. And then read and record the peaks. The average value is calculated by using the formula (1):

$$\overline{\lambda} = \frac{1}{n}\sum_{i=1}^{n}\lambda_i \qquad \cdots\cdots\cdots\cdots (1)$$

λ_i——the measuring value

n ——the measuring times. Here n=3

The difference between the average value $\overline{\lambda}$ and the standard value of three times is the wavelength accuracy. The difference between the maximum value and the minimum value of the three times is wavelength reproducibility.

2) Resolution:

2.1 Testing tools

Hollow cathode lamp: manganese (Mn)

2.2 Testing method

Select 0.2 nm as the bandwidth and 2mA Mn current to search peak at 279.5 nm. Three peaks at 279.5 nm, 279.8 nm, and 280.1 nm can be observed from the spectrogram. And the valley energy values at 279.5 nm and 279.8 nm should be less than specified values.

3) Baseline stability

3.1 Testing tools

Hollow cathode lamp: copper (Cu)

3.2 Testing method

Warm-up the instrument and the Cu lamp for 30 minutes, then select 0.2 nm as the bandwidth and Cu lamp current 3mA to search the peak at 324.7 nm. And then test the sample by flame working mode, set the display range from -0.006Abs to 0.006 Abs. The difference between the maximum Abs and the minimum Abs is the baseline stability.

4) Characteristic concentration

4.1 Testing tools

a. Hallow cathode lamp: copper (Cu)
b. Cu standard solution 0.5 μg/ml
c. Blank solution 0.5% nitric acid solution
d. Air compressor, acetylene cylinder

4.2 Testing method

Adjust the parameters to the optimum conditions, and then alternatively measure the Cu standard solution and blank solution three times. Calculate the characteristic concentration by using the formula (2):

$$C_c = \frac{0.0044 \times C}{\bar{A}} \quad (\mu g / ml / 1\%) \qquad \cdots\cdots\cdots\cdots\cdots\cdots (2)$$

C ----- Concentration of standard solution

$\bar{A}$ ----- Average Abs

5) Detection limit

5.1 Testing tools

a. Hallow cathode lamp: copper (Cu)
b. Blank solution 0.5% nitric acid solution
c. Air compressor
d. Acetylene cylinder

5.2 Testing method

Adjust the parameters to the optimum conditions, set the Abs display to four places of decimals and integration time as 3s, then continuously measure the blank solution for 20 times. Calculate the detection limit by using formula (3):

$$Cl = \frac{3\sigma C}{\bar{A}} \quad (\mu g / ml) \qquad \cdots\cdots\cdots\cdots\cdots\cdots (3)$$

C -----Concentration of the solution

$\bar{A}$ ----- Average Abs

σ ------ standard deviation of blank solution for 20 times

6) Character mass

6.1 Testing tools

a. Hallow cathode lamp: copper (Cu), cadmium (Cd)

b. Standard solution: Cd: 0.001 μg/ml, Cu: 0.1 μg/ml

c. Graphite furnace

d. Pipette

6.2 Testing method

Adjust the parameters to the optimum conditions, set the heating procedure of graphite furnace, inject the solution to the graphite furnace, then measure it for three times. Calculate the character mass by using formula (4):

$$Q_c = \frac{C \times H \times 0.0044}{\overline{A}} \quad (g) \qquad \text{………………………………(4)}$$

H ------ Sample size

C ------ Concentration of the standard solution

$\overline{A}$ ------ The average Abs of three times

7) Precision

7.1 Testing tools

a. Hallow cathode lamp: copper (Cu), cadmium (Cd)

b. Solution can produce 0.3 Abs～0.5 Abs

c. Blank solution 0.5% nitric acid solution

d. Air compressor

e. Acetylene cylinder

f. Graphite furnace

g. Pipette

7.2 Testing method

7.2.1 Flame analysis

Adjust the parameters to the optimum conditions, set the range extend to 1, integration time 3s, Abs between 0.3 Abs～0.5 Abs. Then continuously measure the sample solution and blank solution for 11 times alternatively. Calculate the precision by using formula (5):

$$RSD = \frac{S}{\overline{A}} \times 100\% \qquad \text{………………………………(5)}$$

S ------ Standard deviation

$\overline{A}$ ------ Average Abs

7.2.2 Graphite furnace analysis

Set the heating procedure, choose peak height mode, inject the solution which can produce 0.3 Abs～0.5 Abs into the graphite furnace. Then continuously measure it for seven times.

Calculate the precision by using formula (5):

The user can read the RSD value for precision.

- **The installation acceptance of the instrument conforms to the specifications of the packing list.**

Chapter 3 Instrumental adjustment

Before adjust the instrument, please read “AAWIN” instruction manual carefully. You will learn in this chapter how to make adjustments to the instruments in order to optimize the instrumental conditions for measurement.

Note: AAWIN software will upgrade, so the following pictures may be different from the software in use.

3.1 Selecting the software run mode

When you start up AAWin software, you will see first a starting headline window referring to Figure 3-1. This starting window will soon disappear when you turn on the power for the AA instrument and properly connect the communication cable. If it is not the case, a message box will pop up reminding you to check the connections. When you make sure that all the connections are in normal, click “Retry” for re-communication with the AA main unit. The starting window goes away after the communication with the AA main unit is successfully attached. You can also click “Cancel” directly to enter into the offline mode.

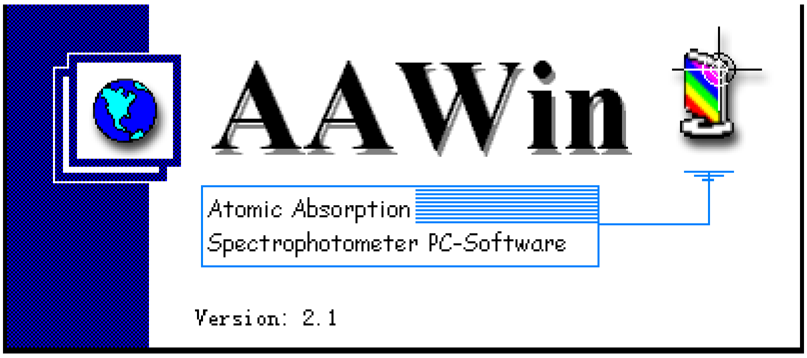


Fig. 3-1 Headline window

A run mode selection box will come up next for you to choose a proper run mode of the software. Refer to figure 3-2. You may check “Online” or “Offline” from the dropdown box for a run mode selection.

- **“Online”**: When you need to make an AA analysis, you can select “Online” and then click “Confirm”, system will switch to the instrumental initialization to set all the initial instrumental parameters.

- **“Offline”**: If you want to get familiar with the AAWin software operations or need to output the former measure results, you may check the “Offline” and click “Confirm”, system will start up in offline mode. In this case, you cannot control the instrument.

- **"Exit":** If you don not want to run the AAWin software, click "Exit" to close the AAWin software system.

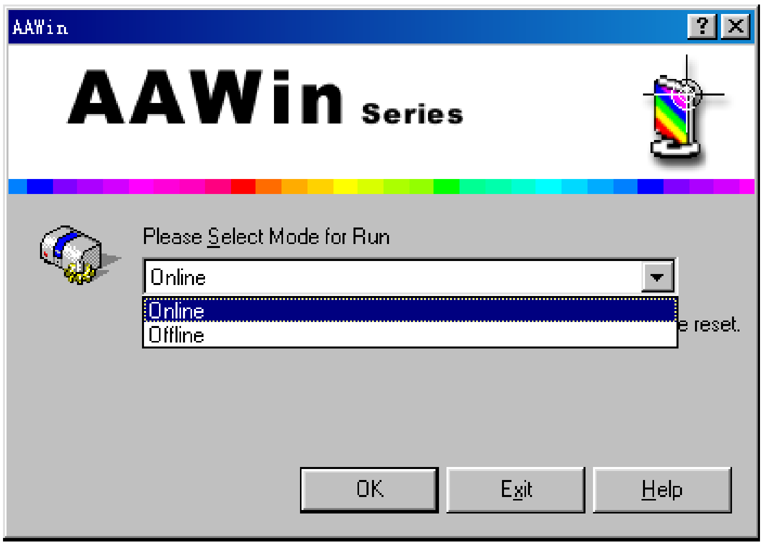
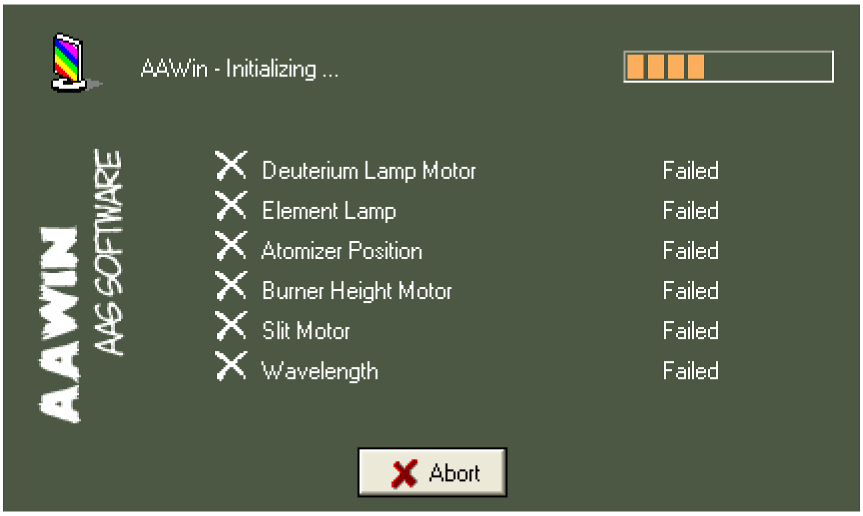


Fig. 3-2 Window for software run mode selection

3.2 Initialization

When you select online mode for running the software, system will initialize the instrument after start-up. See figure 3-3. The instrumental initialization is mainly aimed at the setup of initial parameters of the individual drive motors including D_2 lamp motor, HCL lamp motor, atomizer motor, burner motor, slit width motor and wavelength motor as well. When a certain entry is successfully initialized, a check mark ✓ will come up on the left beside the item; Otherwise, a cross sign ✗ will turn up. If any one of them fails to be successfully initialized, the whole instrumental initialization will not be passed. In this case, a message box will come up after the initialization for you to choose whether you want to try it again or not. You may enter "**Yes**" to continue or press "**No**" to exit the AAWin system. Note that the above message box will pop up only in the online run mode. When you choose [Application]—[Initialization] from the menu to carry out the instrumental initialization, the above message box will not appear.



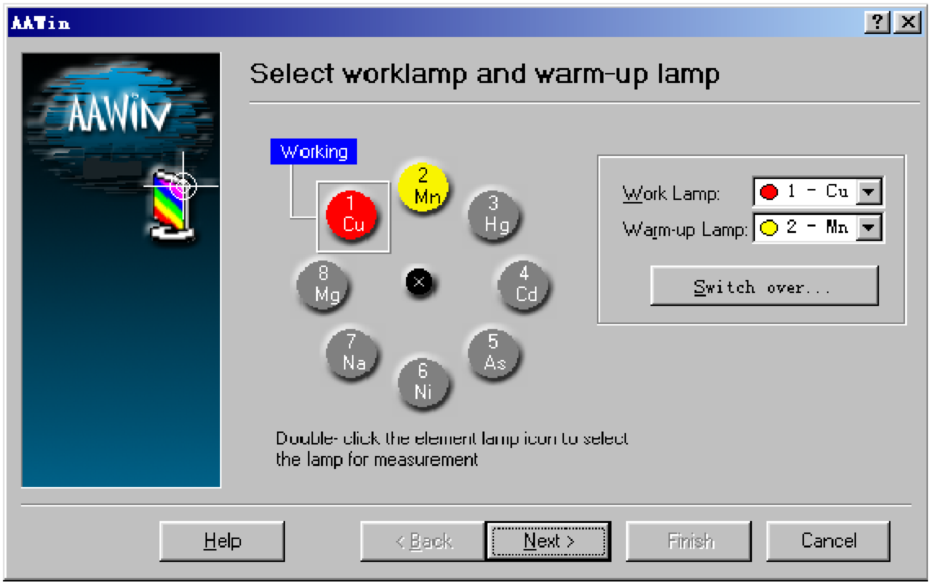
AAWin - Initializing ...

✗ Deuterium Lamp Motor	Failed
✗ Element Lamp	Failed
✗ Atomizer Position	Failed
✗ Burner Height Motor	Failed
✗ Slit Motor	Failed
✗ Wavelength	Failed

Fig. 3-3 Window for initializing the instrument

3.3 Set the element lamp

When you finish the instrumental initialization, system will pop up the element lamp setup guide, which will allow you to make lamp and its element parameter settings stepwise. The guide contains totally four pages, in the first page you are supposed to set the working lamp and the warm-up lamp referring to Figure 3-4. You may select a working or warm-up lamp from its dropdown box respectively, or you can also make exchange of them through the exchange button.



Select worklamp and warm-up lamp

Work Lamp: 1 - Cu

Warm-up Lamp: 2 - Mn

Double-click the element lamp icon to select the lamp for measurement

Fig. 3-4 Selecting the working lamp and preheating lamp

When you need to modify the lamp element, double click the element lamp entering into the element selection dialog box. See Figure 3-5.

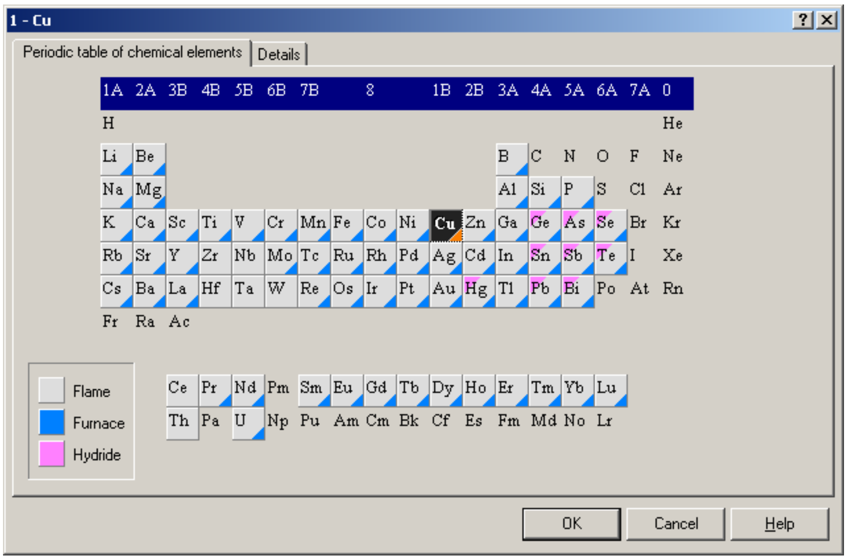
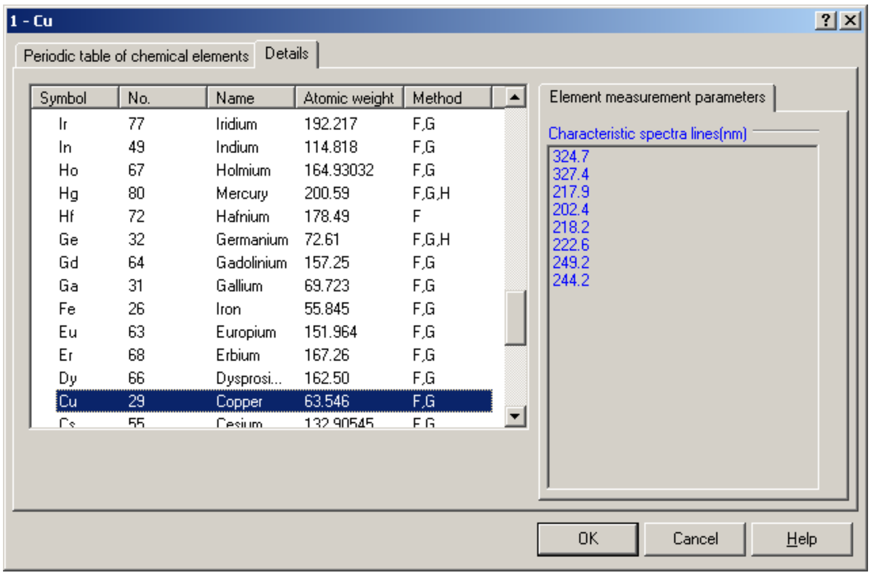


Fig. 3-5 Elementary periodic table

The element selection box has two optional pages in all, the first of which is provided with an elementary periodic table allowing you to make an element selection. In this periodic table elements are marked with different color and designs, which indicates the analytical methods available for the element. All the three AA analytical methods are marked with color, in which the white color “□” stands for the flame method (FA), the green “◢” indicates the furnace method (GFA), and the pink “◤” is for the hydride generation method (HGA). For example, the element tungsten is marked with “W”, showing that tungsten can be measured by flame method; Cupper is labeled with “Cu”, indicating that cupper can be measured by both flame and furnace method; Mercury is marked with “Hg”, which means that all the three AA analytical methods can be used to measure this element. For the element hydrogen, however, there is no mark at all, the color being the same as its background color, which means, none of the three AA methods can be used for this element.

Through marking the elements with color according to the measure method, it will be convenient for you to choose a proper method for the element when you make analysis.

In the second page for the element selection box some detailed information about the elements was displayed in the form of list, providing you with a variety of standard reference conditions for the elements including the specific wavelengths for the element, band width, lamp current, and the fuel flow rate as well. Hence, you can easily choose a proper condition for your measure element from the said expert information system. Refer to Figure 3-6.

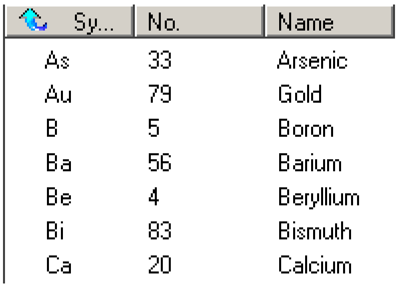


Symbol	No.	Name	Atomic weight	Method
Ir	77	Iridium	192.217	F,G
In	49	Indium	114.818	F,G
Ho	67	Holmium	164.93032	F,G
Hg	80	Mercury	200.59	F,G,H
Hf	72	Hafnium	178.49	F
Ge	32	Germanium	72.61	F,G,H
Gd	64	Gadolinium	157.25	F,G
Ga	31	Gallium	69.723	F,G
Fe	26	Iron	55.845	F,G
Eu	63	Europium	151.964	F,G
Er	68	Erbium	167.26	F,G
Dy	66	Dysprosi...	162.50	F,G
Cu	29	Copper	63.546	F,G
Cs	55	Cesium	132.90545	F,G

Characteristic spectra lines(nm)
324.7
327.4
217.9
202.4
218.2
222.6
249.2
244.2

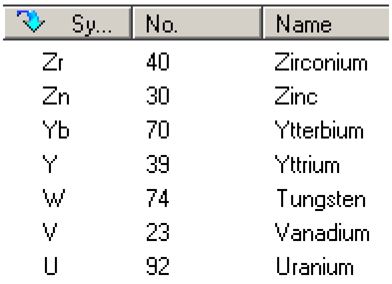
Fig. 3-6 Detailed information about the elements

In this page all the elements which can be measured in AA analysis are listed on the left table, presenting you the element symbol, serial number, name, atomic weight and together with the measure method suitable for the element. In the method column of this table, some abbreviation symbols are used , in which “F” stands for the flame method, “G” shows the furnace method, and “H” indicates the hydride generation method. You may click the headline of each column in the list to sort the data ascendingly or descendingly. See Figure 3-7 and 3-8.



Sy...	No.	Name
As	33	Arsenic
Au	79	Gold
B	5	Boron
Ba	56	Barium
Be	4	Beryllium
Bi	83	Bismuth
Ca	20	Calcium

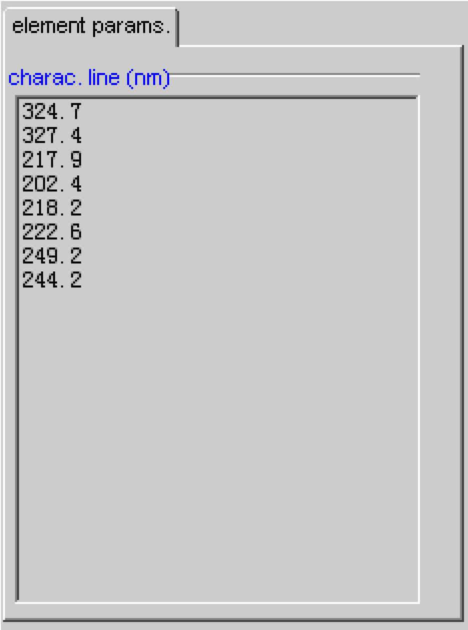
Fig. 3-7 Sorted by element symbol ascendingly



Sy...	No.	Name
Zr	40	Zirconium
Zn	30	Zinc
Yb	70	Ytterbium
Y	39	Yttrium
W	74	Tungsten
V	23	Vanadium
U	92	Uranium

Figure 3-8 Sorted by element symbol descendingly

Below the element list table on the left will be provided with the description of related measure methods available to the element selected in the list. For example, when the element cupper (Cu) was highlighted in the list, the following message will be displayed: “Flame: air—acetylene, oxidative (blue)”. On the right of element list table are the characteristic spectral lines of the selected elements. The user can use scrollbar to read. See fig.3-9.

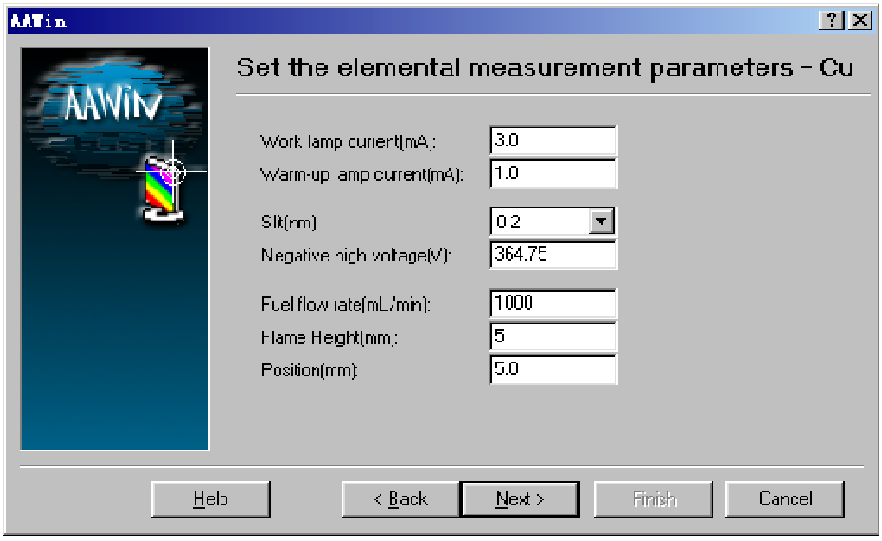


charac. line (nm)

324.7
327.4
217.9
202.4
218.2
222.6
249.2
244.2

Fig. 3-9 Characteristic spectral lines

When you finish selecting the elements, click “Confirm”. The system will allow you to modify the element you just selected on the current lamp position. In this case, you may click “Next” entering into the element lamp parameter setup page. See Fig. 3-10.

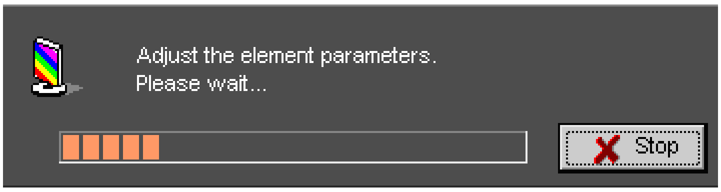


Set the elemental measurement parameters - Cu

Work lamp current(mA): 3.0
Warm-up lamp current(mA): 1.0
Slit(nm) 0.2
Negative high voltage(V): 364.75
Fuel flow rate(mL/min): 1000
Flame Height(mm): 5
Position(mm): 5.0

Fig. 3-10 Set measurement parameters

In this page, you can set the lamp parameters for the selected element, which will include working current, warm-up current, bandwidth and the negative high-voltage. Both the working current of the lamp and the bandwidth are taken from the database of the expert information system as the default setting values. You may also enter other values if you do not want to use the default ones. Click “Next”, system will make adjustment to the instrument according to your settings. Refer to Figure 3-11.

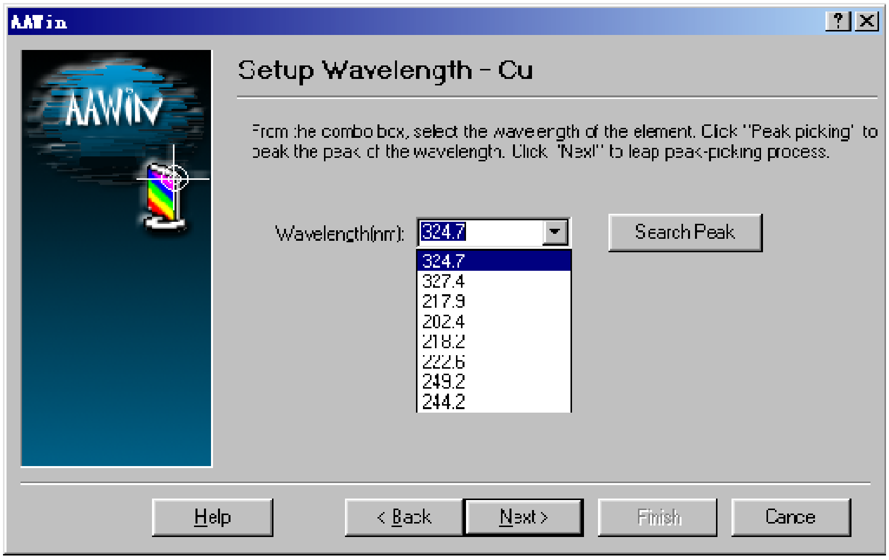


Adjust the element parameters.
Please wait...

Fig. 3-11 Adjusting the element parameters

Note: This window will always be applied in AAWin software system, when needed to deliver the control parameters for the instrument. You can click “Cancel” to quit from the execution of this instruction.

After you finish the above settings, you may switch to the wavelength setup page. In this page, you can choose the characteristic wavelength for the specified element and perform the peak searching referring to Figure 3-12.



Setup Wavelength - Cu

From the combo box, select the wavelength of the element. Click "Peak picking" to peak the peak of the wavelength. Click "Next" to leap peak-picking process.

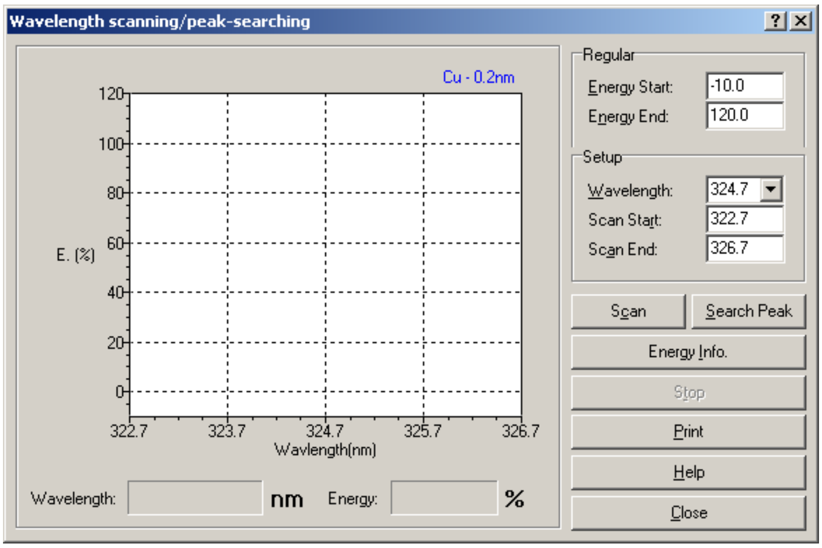
Wavelength(nm): 324.7

Fig. 3-12 Setting wavelength

Select a resonance line from the dropdown box of wavelength, and click “Peak-search” to perform the peak searching (For detailed information, see Section 3.4 Peak search and wavelength scan). When peak search is complete, you will end up the element lamp setup. Click “Finish” to close the setup guide. If you did not perform the peak-search, system will remind you to do so when you click the “Next” button. As you have already changed the parameters for the element lamp, the optimal position of the wavelength may also have changed, therefore a peak search is recommended in this case. You can choose “Yes” for peak search or “No” to skip. If you do not need reminding, click “Cancel”.

3.4 Peak search and wavelength scan

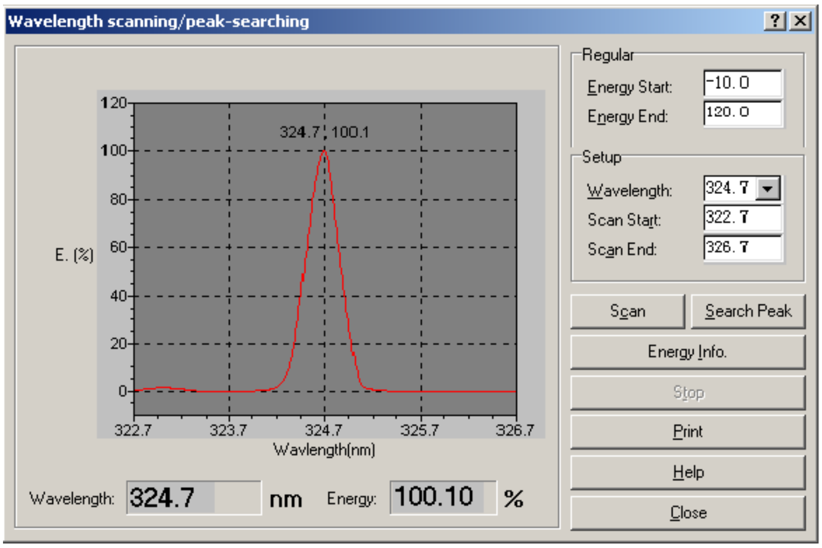
After you are complete in the element lamp setup, you can carry out peak search and scan the wavelength profile. Select [Application]—[Wavelength scan / Peak-search] from the menu or click the “ ” button from the toolbar to open the wavelength scan and peak search dialog box. See Figure 3-13.



Energy Start: -10.0
Energy End: 120.0
Wavelength: 324.7
Scan Start: 322.7
Scan End: 326.7

Fig. 3-13 Wavelength scan and peak-search window

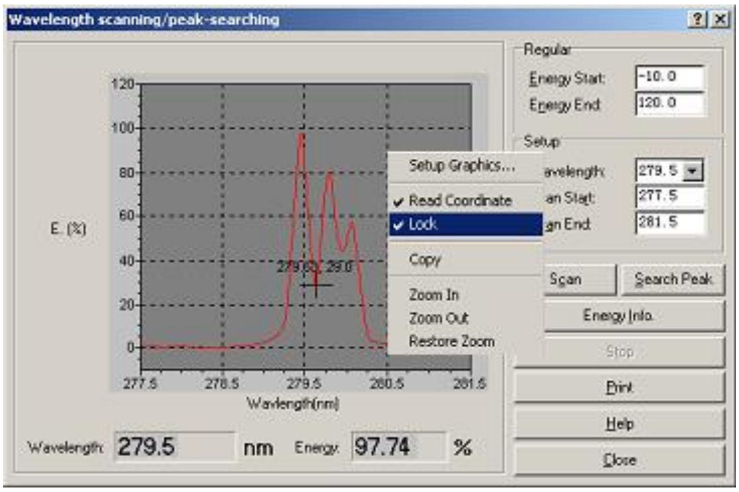
Click the “Peak search ” button in this box to set the peak position of the current wavelength. If you need to make peak search for other characteristic wavelengths of the specified element, select the corresponding wavelength from the dropdown box and click the “Peak search” button. The result for peak search will be dynamically displayed on the wavelength scan graph, and the maximal energy position (Peak position) will be sorted out and displayed below the wavelength scan graph after the fine adjustment to the instrumental energy. See Figure 3-14. If you want to check the current energy state of the instrument, click the “Energy” button to open the energy adjustment window. Refer to Section 3.6 for more information.



Energy Start: -10.0
Energy End: 120.0
Wavelength: 324.7
Scan Start: 322.7
Scan End: 326.7
Wavelength: 324.7 nm Energy: 100.10 %

Fig. 3-14 Display of peak-search result

Sometimes you need to scan a certain range of wavelength in order to get more detailed information about the wavelength profile within that range. In this case, you may enter the starting point and end point of the wavelength separately in each of the input box, and input the lower and upper limits of the energy (the vertical coordinate axis) in the respective input box, then click “Scan” to perform wavelength scanning within the specified range. Note: The values of the wavelength should be at the range of 190~900nm. If you entered a value out of that range, system will pop up an error message and allow you to enter it again. After finished, you can also read out the coordinates of the peak profile graph. Right click on the graph, select [Coordinate readout] from the popup menu to get the coordinates of peak position. If you need to lock the readout coordinates, check the “Lock” option from the above popup menu. To unlock the readout coordinates, re-check the “Lock” option. Refer to Figure 3-15.



Energy Start: -10.0
Energy End: 120.0
Wavelength: 279.5
Scan Start: 277.5
Scan End: 281.5
Wavelength: 279.5 nm Energy: 97.74 %

Fig 3-15 Lock the coordinate range

If you want to terminate the instrumental operation in the process of wavelength scan or peak search, click the “Stop” button to quit from the current operation. Note: Abnormal termination of the instrument may affect the accuracy of measurement. Hence, this function will here not be recommended.

The peak search or wavelength scan graph will be preserved by the AAWin system in order to be examined when needed. You may also click “Print” to make it a print out. The scan graph will not be removed until you perform the peak search or wavelength scanning for a second time. In that case, a new scan graph will turn up to substitute the old one.

3.5 Wavelength correction

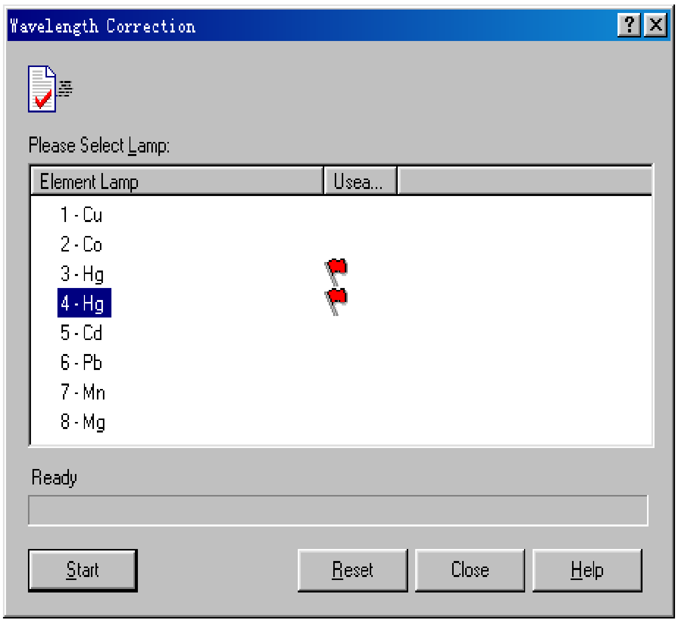
If you have got a large wavelength error (more than ±0.3nm) while you perform the peak search, you may use the wavelength correction function to calibrate the wavelength.. To do so, select [Application]—[Wavelength correction] from the menu to open the wavelength correction box. Refer to Figure 3-16.

You need first to choose the turret number of the lamp used for wavelength correction from the element lamp setup list. Note: Only the mercury element lamp (Hg) can be applied to the wavelength correction. Therefore, the mercury lamp has been specially labeled with “🚩” in the elementary lamp list. You are only allowed to perform the wavelength correction with the labeled element lamp; otherwise, the wavelength correction will not proceed. You can use the element lamp setup guide to set the lamp parameters for the element (See Section3.3 Set the element lamp).

After you have selected the element lamp, click “Start” to perform the wavelength correction. If you want to terminate the operation while the wavelength correction proceeds, you may click the “Stop” button. See Figure 3-17. If an error massage appeared during the correction process as follows: “Wavelength error is too large, correction can not proceed”, it indicates that the wavelength error of the instrument is out of the limitation range or the AA instrument is not in a normal state. In this case, you may check if the atomizer or the burner head is properly set on the AA optical path or if they are blocking the light path. You need to re-set the position of the burner head or of the atomizer in the case of light blocking until the light can pass through smoothly. Click “Reset” to restore the initial parameters of the wavelength setup, and then perform the wavelength correction again. If the error message remains to come up, you need to readjust the optical path of the instrument. In that case, please contact with your representative in the local place for repair.

In general, a frequent wavelength correction is unnecessary and should therefore be avoided. Only in the following conditions is a wavelength correction required:

1. After a new AA instrument is installed;
2. The physical location for the AA instrument is changed;
3. The environment conditions are largely changed.

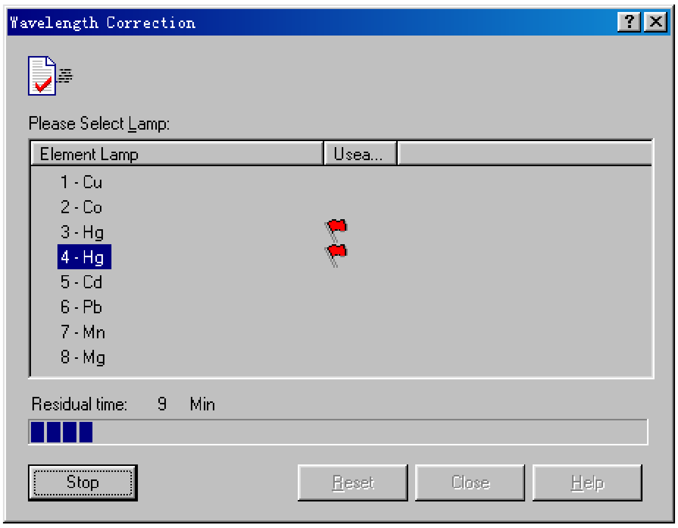


Please Select Lamp:

Element Lamp	Usea...
1 - Cu	
2 - Co	
3 - Hg	⚑
4 - Hg	⚑
5 - Cd	
6 - Pb	
7 - Mn	
8 - Mg	

Ready

Figure 3-16 Wavelength correction window



Please Select Lamp:

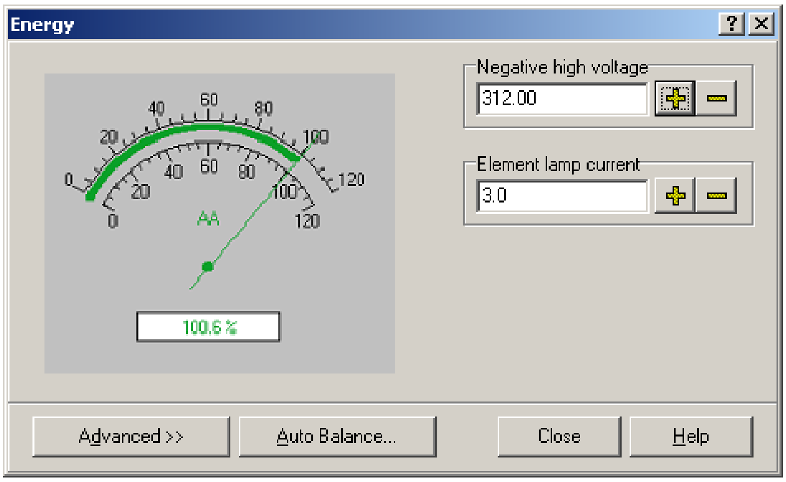
Element Lamp	Usea...
1 - Cu	
2 - Co	
3 - Hg	⚑
4 - Hg	⚑
5 - Cd	
6 - Pb	
7 - Mn	
8 - Mg	

Residual time: 9 Min

Figure 3-17 Running the wavelength correction

3.6 Energy adjustment

When you need to check the energy state of the instrument or make an adjustment of energy, select [Application]—[Energy adjustment] from the menu or click the “ ” button from the toolbar to open the energy adjustment box, see Figure 3-18. In this box, a sector (fan-shaped) plate meter with different color needle indicators is used to indicate the current energy state of the AA and BG signals, in which the green color shows the AA energy signal, and the red color stands for the BG energy signal. If no BG correction mode is used, the green energy indicator is only displayed. You can enter a value for the negative high voltage in the input box (within the range of 0~1000V) followed by a “Enter” key to make an adjustment of the negative high voltage, or press the “+” or “−” button to increase or decrease the high voltage value with the amplitude of 0.25 V for each step. When you need to re-adjust the lamp current, you can enter a current value (within the range of 0~20mA) in the lamp current input box followed by a “Enter” key， or use the “+” or “−” button to make a fine adjustment with the amplitude of 0.1mA for each step. Use the same method as above to adjust the D_2 lamp current or the wide and narrow pulse currents, when the D_2 lamp or SR lamp BG mode is selected. Refer to Figures 3-19 and 3-20.

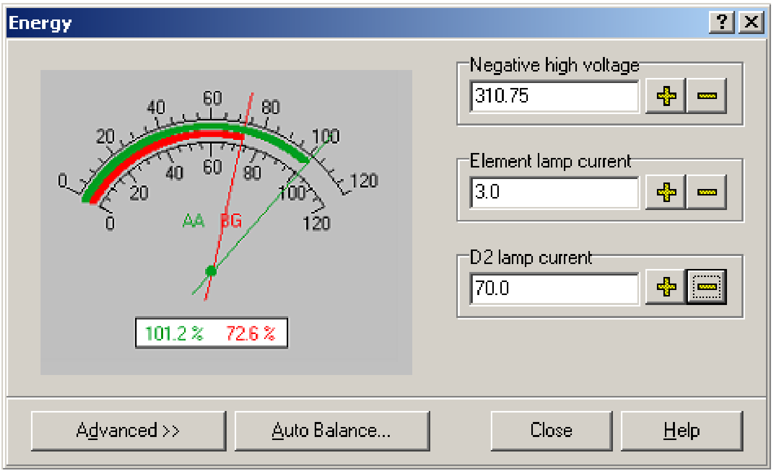


Negative high voltage: 312.00

Element lamp current: 3.0

100.6 %

Fig. 3-18 Energy adjustment window



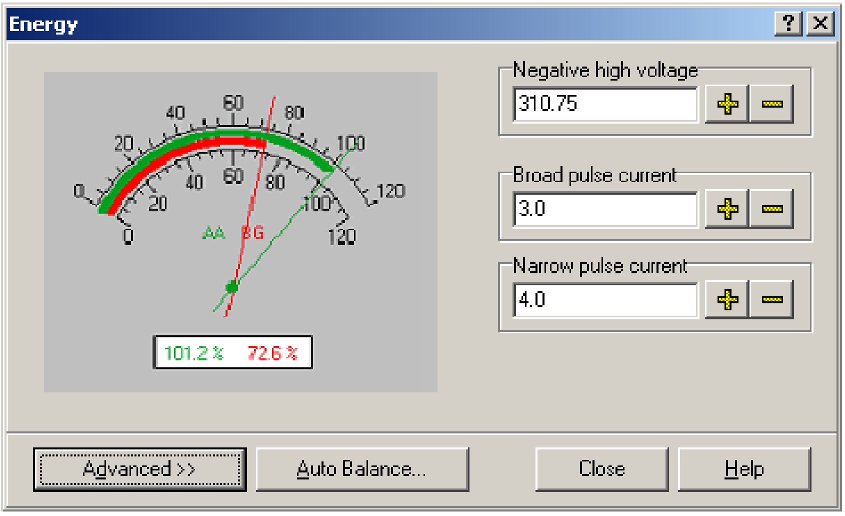
Negative high voltage: 310.75

Element lamp current: 3.0

D2 lamp current: 70.0

101.2 % 72.6 %

Fig. 3-19 D2 lamp energy adjustment



Negative high voltage: 310.75

Broad pulse current: 3.0

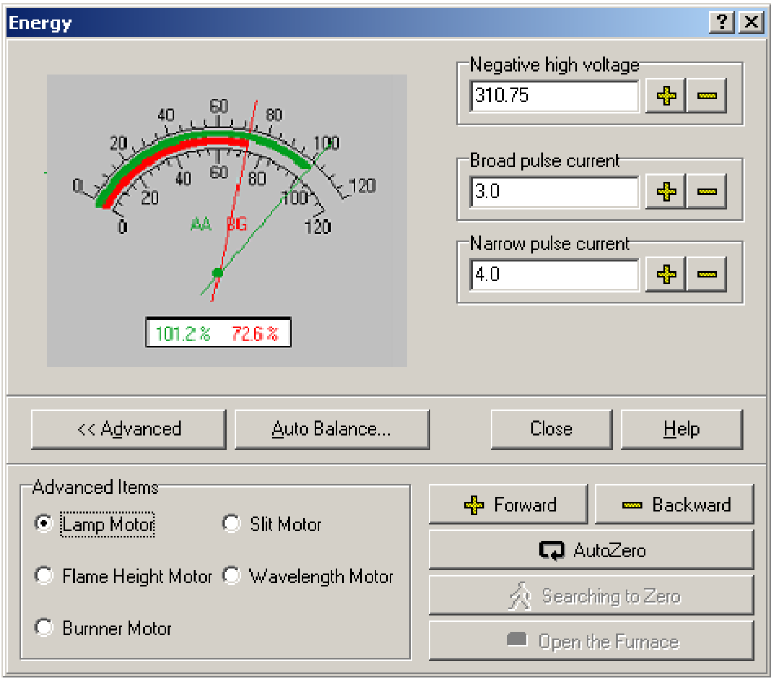
Narrow pulse current: 4.0

Fig. 3-20 SR lamp energy adjustment

If you cannot perform a successful beam balance manually, click the "Auto beam balance" button for an automatic adjustment. In this case, system will automatically adjust the instrument to the beam balance state, providing you with a great convenience.

You can also make an advanced adjustment to the instrumental parts in the energy adjustment box. Click "Advanced adjustment" to open the advanced adjustment box, see Figure 3-21. Select one entry from the advanced adjustment option box, use the four buttons on the lower right of the box to make adjustment by step. For the "burner motor" and the "slit motor", no zeroing can be performed, and the "zero-order spectra line search" is the unique function for the "wavelength motor". If you select D2 lamp as a background correction, the D2 lamp mirror motor can also become active in the advanced adjustment box. You may use the "positive rotation" or "Reverse/Back turn" button to adjust the light path of the D2 lamp until the beam spot of the D2 lamp becomes fully overlapped with that of the element lamp.

Note: The advanced adjustment function should be of limited use in the practical work of measurement in case that the instrumental parameters may become in disorder. When there is no special need, the frequent use of this function should be desirably avoided.



Negative high voltage: 310.75

Broad pulse current: 3.0

Narrow pulse current: 4.0

Advanced Items

● Lamp Motor ○ Slit Motor

○ Flame Height Motor ○ Wavelength Motor

○ Burnner Motor

Fig. 3-21 Advanced adjustment

3.7 Set the instrumental parameters

In this section, we will introduce you how to set the parameters for the instrumental hardware portions. As these parameters are tightly (closely) related to the hardware driving system of the instrument, any negligence or carelessness in the parameter settings may cause a damage of the instrument, users are assumed to carefully read the following sections and become familiar with their operations before performing the parameter settings.

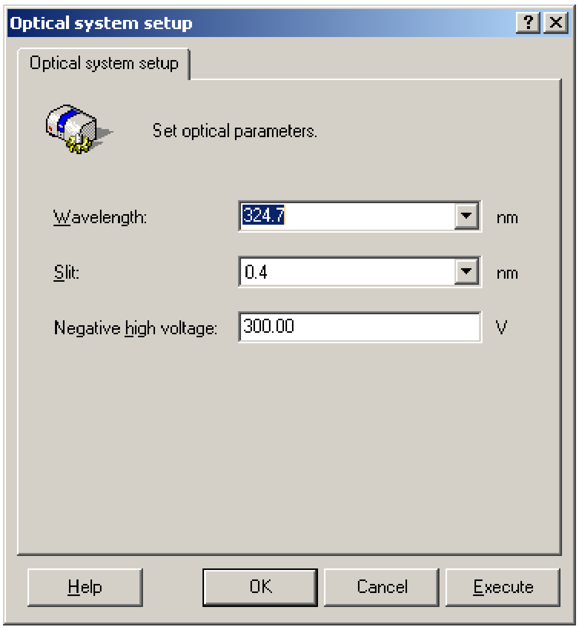
3.7.1 Set optical parameters

When you need to set the working wavelength, bandwidth and the negative high voltage, select [Instrument]—[Optical system] from the menu to open the optical parameter setup box, refer to Figure 3-22. You can select the working wavelength from the wavelength dropdown box or enter the value of the current working wavelength in the input box. The wavelength you entered should be at the range of 190~900nm, If not， system will pop up a message box for warning.

You may choose the bandwidth from the dropdown box. The bandwidth available is as follows: 2.0nm, 1.0nm, 0.4nm, 0.2nm and 0.1nm.

You can enter the current negative high voltage in the high voltage input box, the value of which should be at the range of 0~1000V. If it is out of the range, system will give you a warning message.

After you finish the regular parameter settings, click “Determine” for the system to transfer your settings to the AA instrument. In the mean time, the instrumental parts will be adjusted according to your settings. When the adjustment is over, system will close the above dialog box. If you do not want to close the dialog box, you my click “Execute” instead of “Determine”. In this case, system can also deliver your settings to the instrument, but do not close the dialog box after adjustment, allowing you to make a further adjustment to the instrument.



Set optical parameters.

Wavelength: 324.7 nm

Slit: 0.4 nm

Negative high voltage: 300.00 V

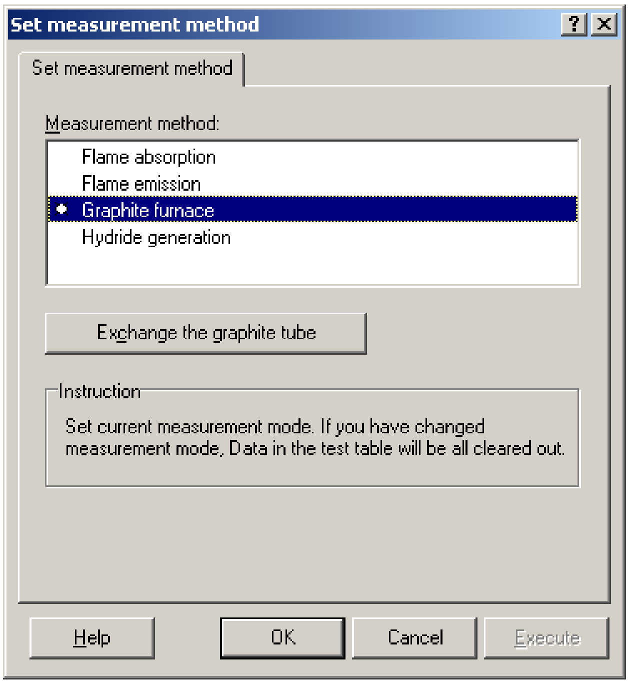
Fig. 3-22 Optical parameters setup window

3.7.2 Set the measure method

When you need to modify your current measure method, select [Instrument]—[measure method] from the menu to open the measure mode setup box, see Figure 5-22. Before doing so, you should make sure that all the measure results in the measure table have been properly preserved or printed out in case of any lost, because all the data in the measure table will be removed once you have changed your current measure method.

You can set the measure method from the dialog box according to your measure requirements. The methods that are available in the box are listed as follows: flame absorption, flame emission, graphite furnace atomization and hydride generation method etc. For example, if you need to change your current measure mode from flame to furnace method and exchange the graphite tube afterwards, you can select “Graphite Furnace” from the “Measure method” list box, and then click “Execute”. After the switchover is complete, click the “Exchange the graphite tube” button to open the furnace body, allowing you to replace a new graphite tube. （If you use the manual exchange instrument, you need to change from flame analysis to graphite furnace manually. Pull out the exchange platform to the end is ok.）After you finish the replacement, click [Determine] to close the furnace body.

Note: Before switching to the Graphite furnace mode, remove the stop plate between the burner head and the furnace body, lift up the sample support stand and the combustion chamber door in case of any damage in the instrument during the process.



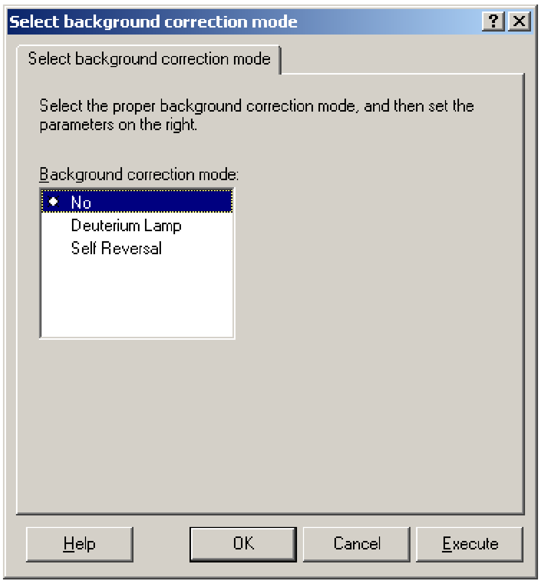
Measurement method: ● Graphite furnace

Set current measurement mode. If you have changed measurement mode, Data in the test table will be all cleared out.

Fig. 3-23 Measure method selection window

3.7.3 Set the background correction mode

In the measurement of atomic absorption analysis, if the bulk of the measured sample is too complicate, which gives a large background absorption, causing a serious problem of spectral interference, or the content of the element to be measured is too low, where high sensitivity is required and the atomic absorption signal need to be more accurately corrected, in this case, you need to use the background correction mode for measurement. The background correction modes that are available for AA-990 series atomic absorption spectrophotometer are as follows: D_2 lamp-BGC and SR lamp-BGC mode. You can select [Instrument] — [Background correction mode] from the menu to open the background correction mode selection box, refer to Figure 3-24.



Select background correction mode

Select background correction mode

Select the proper background correction mode, and then set the parameters on the right.

Background correction mode:

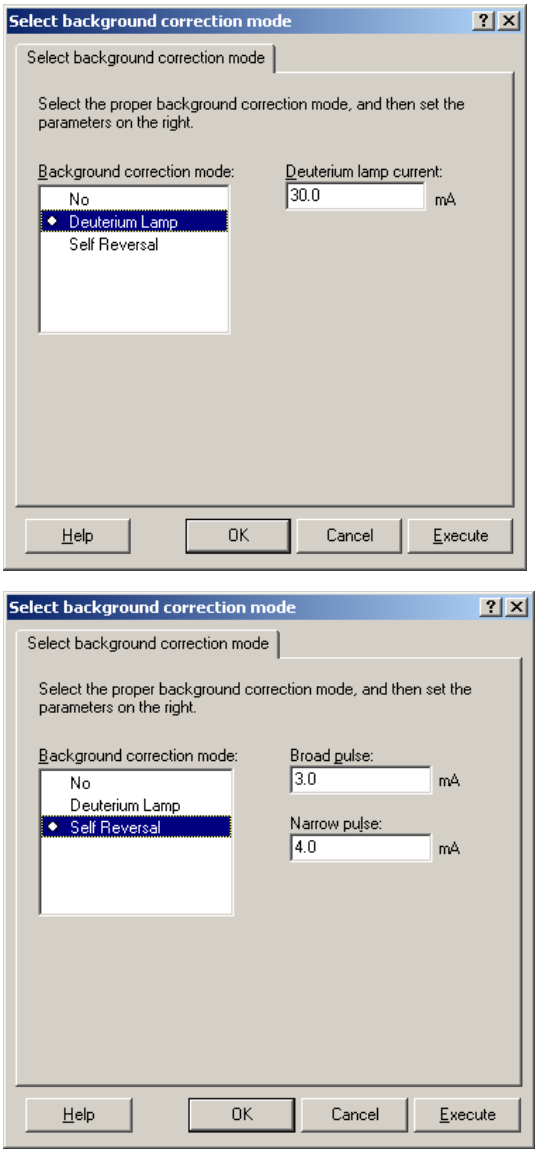
- No
- Deuterium Lamp
- Self Reversal

Fig. 3-24 Selecting background correction

You can choose the background correction mode from the list box and click [Determine] or [Execute], system will immediately set the background correction mode you selected as the current mode. If you select the D_2 lamp to perform the background correction, an input box for D_2 lamp current will pop up on the right side of the BGC mode list box, allowing you to set the lamp current values according to your own requirements. When you choose SR lamp to perform the background correction, there will be two input boxes coming up on the right of the BGC mode list box, allowing you to enter the wide and narrow pulse current values for the SR lamp. Refer to Figure 3-25.

Based on the measured elements, the default values for the D_2 lamp current or the SR lamp currents (the wide and narrow pulse current) may be different, which are all taken from the database of the expert information system. When you set the D_2 lamp current, the value you entered should be at the range of 0~120mA. When set the SR lamp currents, the value of the wide pulse current should be controlled within 0~20mA, and the narrow pulse current within 0~10mA.If the values you entered are out of the limitation range, system will pop up an error message for warning.

Note: When D_2 lamp is used to perform the background correction, it should be always avoided that D_2 lamp is lit up under its maximum power (lamp current =120mA) for long time. When not used for a long period, shut down the lamp power as soon as possible in order to extend its useful life period.



Select background correction mode

Select background correction mode

Select the proper background correction mode, and then set the parameters on the right.

Background correction mode: Deuterium Lamp

Deuterium lamp current: 30.0 mA

Select background correction mode

Select background correction mode

Select the proper background correction mode, and then set the parameters on the right.

Background correction mode: Self Reversal

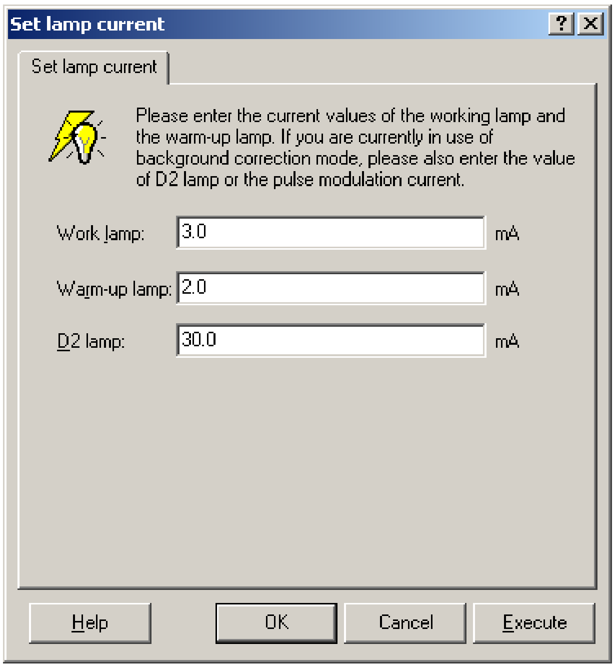
Broad pulse: 3.0 mA

Narrow pulse: 4.0 mA

Fig. 3-25 Set D2 lamp and the SR lamp background

3.7.4 Set the lamp current

When you need to change the working lamp and/or the warm-up lamp current, Select [Instrument]—[Lamp current] from the menu to open the lamp current setup box, see Figure 3-26. You can enter the current values for the working lamp and/or the warm-up lamp in the respective input box, and click [Determine] or [Execute] to transfer your settings. The current value you entered should be controlled within 0~20mA. If it is out of this limitation range, system will pop up a warning message. If you are now using the D_2 lamp to perform the background correction, you can also enter a current value for the D_2 lamp in the D_2 lamp current box.



Please enter the current values of the working lamp and the warm-up lamp. If you are currently in use of background correction mode, please also enter the value of D2 lamp or the pulse modulation current.

Work lamp: 3.0 mA

Warm-up lamp: 2.0 mA

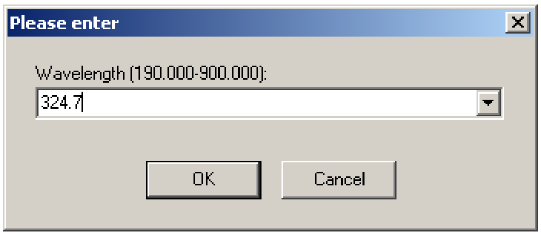
D2 lamp: 30.0 mA

Fig. 3-26 Setting the lamp current

3.7.5 Locating the wavelength

When you need to set the instrumental wavelength to a certain value, you may use the “wavelength location” function. Select [Instrument]—[Wavelength location] from the menu or press F3 key to open the wavelength location window, refer to Figure 3-27. You can choose the wavelength value from the dropdown box or directly enter it into the wavelength box, and then click [determine] to perform.

The analysis of some elements must use the function of wavelength locating, such as Cr analysis. First the user should search the peaks to get peak-searching spectrum, then read the wavelength of the second peak by using coordinate readout. And then locate the wavelength to the main sensitive line. See 3.4 for peak searching and wavelength scan.



Wavelength (190.000-900.000): 324.7

Fig. 3-27 Input box for wavelength location

3.8 Set the burner parameter

When you are performing measurement in Flame mode (Flame absorption, Flame emission and Hydride generation), you need to set the burner parameters.

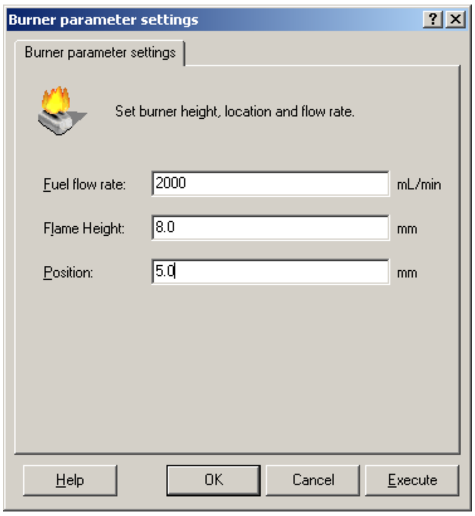
Select [Instrument]—[Set the burner parameters] from the menu to open the burner setup box, see Figure 3-28. You can set the parameters as fuel gas flow rate, burner height and burn position etc in the burner setup box.

Fuel gas flow rate refers to the current flow rate for the combustion gas, which takes ml /min as its unit. Its range of value should be controlled within 1000~3000ml/min. After ignition, system allows you to set the fuel flow for adjustment. In this case, you can observe a dynamic change in the flow rate from the flame.

Burner height can be defined by the vertical distance between the burner head and the center light path for the element lamp, which should be at the range of 0~20mm. **Burner position** can be regarded as the longitudinal separation of the burner head from the light path of the element lamp (namely the optical axis), the value of which should be controlled within -5~5mm.

(The setting is only effective to auto exchange instrument. For the flame analysis instrument, manual adjustment is used. For the manual exchange instrument, tools are used to adjust the burner position.)

When you finish the burner parameter setup and click [Determine] or [Execute] to activate your settings, system will make adjustments to the burner head according to your setup. If there is any value you entered out of the limitation range, system will pop up a warning message.



Set burner height, location and flow rate.

Fuel flow rate: 2000 mL/min

Flame Height: 8.0 mm

Position: 5.0 mm

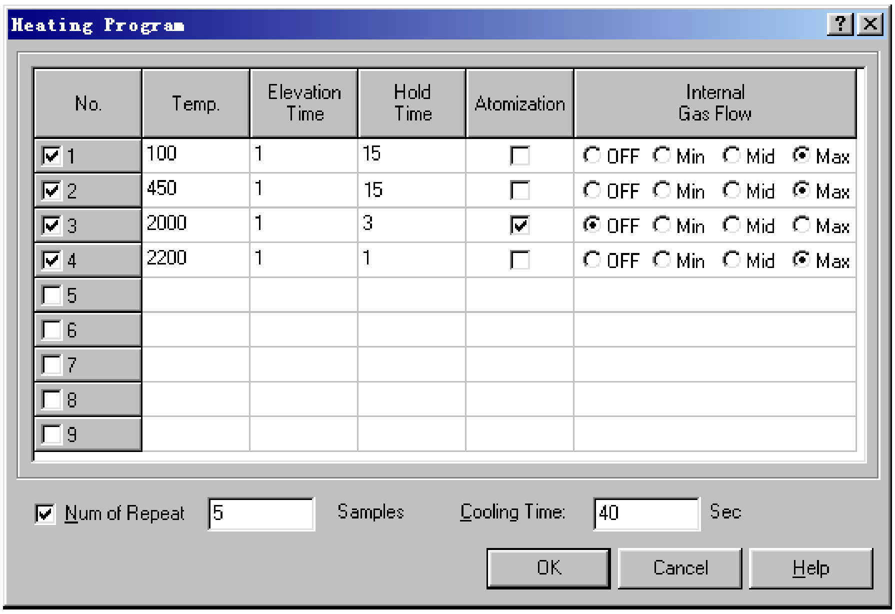
Fig. 3-28 Set the burner parameters

3.9 Set the furnace parameters

When you are using furnace mode to perform measurement, you have to set the furnace parameters, including the furnace heat program parameters as well as the position of the furnace atomizer.

3.9.1 Set the furnace heat program

Before measuring in furnace mode, you need to set the heat program for the furnace. Select [**Instrument**]—[**Set furnace heat program**] from the menu to open the furnace heat program setup box, see Figure 3-29.



No.	Temp.	Elevation Time	Hold Time	Atomization	Internal Gas Flow
☑ 1	100	1	15	☐	○ OFF ○ Min ○ Mid ● Max
☑ 2	450	1	15	☐	○ OFF ○ Min ○ Mid ● Max
☑ 3	2000	1	3	☑	● OFF ○ Min ○ Mid ○ Max
☑ 4	2200	1	1	☐	○ OFF ○ Min ○ Mid ● Max
☐ 5					
☐ 6					
☐ 7					
☐ 8					
☐ 9					

☑ Num of Repeat 5 Samples Cooling Time: 40 Sec

Figure 3-29 Set the furnace temperature program

In this dialog box you are allowed to set a maximum of 9 stages of temperature conditions for the furnace temperature program. Click the check box beside each serial number of stage in the serial number column to activate the stage to perform the heat program setup.

- **"Target temperature"** is the set value of the end temperature for the current stage, which should be controlled within the range of 20~2700℃.
- **"Heating time"** refers to the time period taken by the heat stage in which the furnace temperature is elevated from the set value in the former stage to the target in the current stage. The value for the **heating time** should be at the range of 0~255 seconds.
- **"Hold time"** is defined by the time period during which the furnace temperature is set to keep constant after elevated to the target temperature. The value for the **hold time** should be also controlled within the range of 0~255 seconds.
- **"Atomization"** is used to set the atomizing stage for the furnace heat program in order for the system to perform data sampling in that stage. You can click the check box in the step, which you need to select as the atomizing stage, to activate the atomization setup. Please note that this "**Atomization**" function can be set during only one stage for the heat program.
- **"Inner gas flow rate"** allows you to regulate the gas flow passing through the inside of the graphite tube during each stage. It adopts a gas line system with "**none**", "**low**", "**medium**" and "**high**" four-level flow control. The "**none**" level is mainly used for the atomizing step, during which the inner gas flow rate is totally cut off. The "**Low**" level refers to the gas flow state where the inner gas flow rate is set to a low value with about 150mL/min. For the "**Medium**" level, a middle inner gas flow rate with about 300mL/min has been regulated.

With the “**High**” level inner gas flow rate, approximately a 450mL/min gas flow rate is allowed to pass through the inside of the graphite tube.

Note：When a furnace temperature is set on 2000~2500 ℃, the total time used for heating and hold in this stage should be less than 25 seconds. If a 2500~2700 ℃ furnace temperature is selected, the total time for heating and hold should be controlled no more than 15 seconds. If the value is out of the limitation range, system will pop up a warning message. Heat steps to be set in a furnace program should be logical and coherent, no blank stage is allowed in the furnace program, and moreover the first step in the furnace heat program box must be also selected.

If you need to modify the cooling time for the furnace body, you can enter the value in the input box of the “cool-down time”， which should be within the range of 20~99seconds.

After you finish setting the heat program, click [Determine] to transfer your settings to the instrument and exit the setup window.

3.9.2 Set the furnace atomizer position

When performing measurements in furnace mode, a problem for the offset of the atomizer may sometimes occur, causing the light path from the element lamp not to pass through the center for the graphite furnace atomizer. In this case, you need to re-set the furnace atomizer position. Select [**Instrument**]—[**Set the atomizer position**] from the menu to open the atomizer position setup box, refer to Figure 3-30.

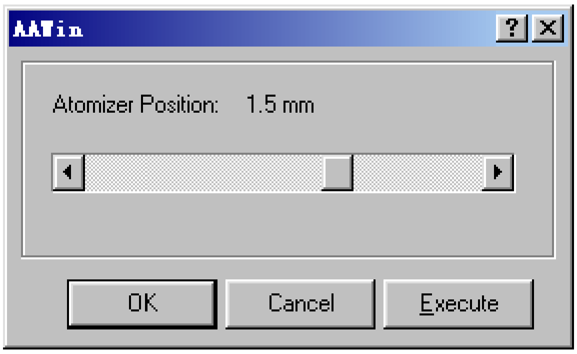

Atomizer Position: 1.5 mm

Fig. 3-30 Set the atomizer position

You can use the left mouse key to drag the scroll bar for adjustment. Once you have set the scroll bar to a proper position, click [**Determine**] or [**Execute**] to activate your setting.

(The setting is only effective to auto exchange instrument. For the flame analysis instrument, manual adjustment is used. For the manual exchange instrument, tools are used to adjust the burner position.)

Through the introduction of this chapter, you are sure to get a better understanding about the adjustments of our AA-990 series AA Spectrophotometer. You may use the methods introduced above to optimize your instrumental conditions before measurement.

Chapter 4 Measurement

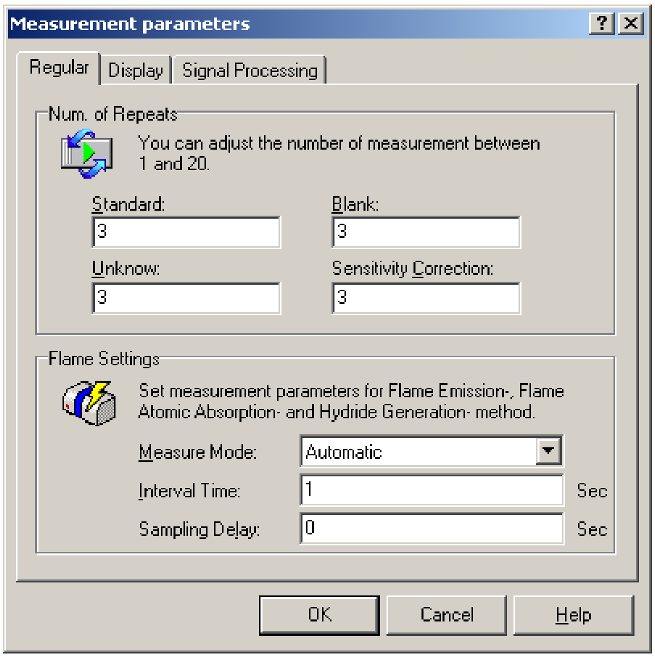
In the preceding chapter we introduced you some basic method and technical know-hows about the adjustment of the instrument. Now you are preparing to make a measurement. In this chapter we will present you how to set measurement parameters and make unknown sample settings, and will here also provide the whole process for making one measurement.

Note: Windows system is multi-tasks system, which allows you run multiply application programs at the same time, but there must be interference between among programs, and the running speed must be slowed down when running multi-programs. So it is recommended that when you measure, you'd better not run other application programs, such as audio program, video program, screen saver and other programs occupying much of CPU.

4.1 Set measurement parameters

To make a measurement, you need to set the measurement parameters first. Select [Settings] — [Measurement parameters] from the menu or click the button from the toolbar, and you will open a dialog box for measurement parameter settings. From the dialog box you will see three optional pages, the first page allows you to make regular parameter settings. See Figure 4-1.

4.1.1 Set regular measurement parameters



Num. of Repeats

You can adjust the number of measurement between 1 and 20.

Standard: 3

Blank: 3

Unknow: 3

Sensitivity Correction: 3

Flame Settings

Set measurement parameters for Flame Emission-, Flame Atomic Absorption- and Hydride Generation- method.

Measure Mode: Automatic

Interval Time: 1 Sec

Sampling Delay: 0 Sec

Fig. 4-1 Setting regular measurement parameters

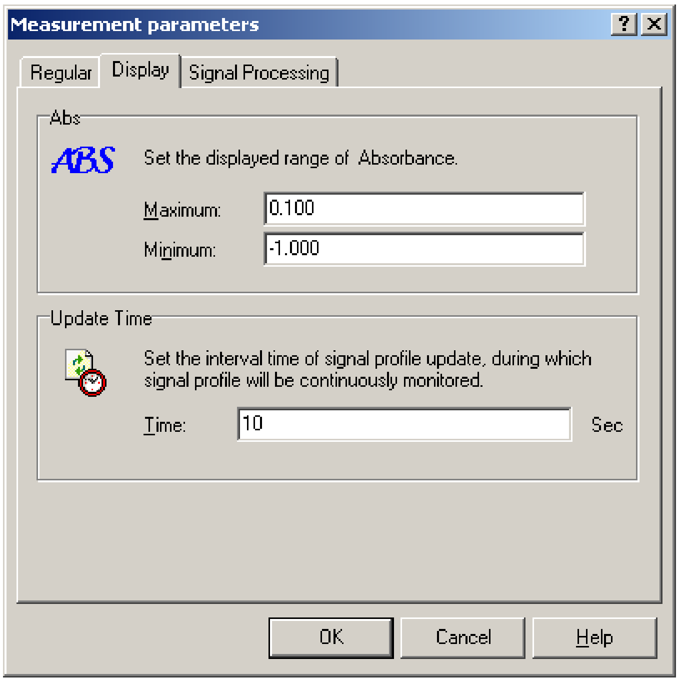
You may set duplicate numbers for the “Standard”, “Unknown”, “Blank” and the “Sensitivity correction sample” in the “Repeat number for measurement” section, the value of which should be at the range of 1 to 20. If the current analytical method you select is Flame Analysis (Flame absorption, Flame emission, Hydride generation method), you can also make some special settings for the Flame Analysis: You can select “Manual” or “Automatic” for the “Measurement mode” to make a measurement. In the “Manual” mode, you need to operate by hand to make every individual measurement. Hence, when a “Manual” mode is selected, the other two options in this section (“Time interval” and “Sampling delayed”) will become inactive; in the “Automatic” mode, system will repeatedly take the measuring process and automatically make all the measurements, users need not to operate by hand during the measuring process. If the “Automatic” mode is chosen, then the other two options—“Time interval” and “Sampling delayed” need also to be set.

“**Time interval**” is the waiting time for each sample uptake; it should be within the range of 1 to 99 seconds.

“**Sampling delayed**” is the waiting time taken before auto-sampling, i.e. the pre-spraying time. It should also be at the range of 1 to 99 seconds.

4.1.2 Display setup

After setting the regular parameters, you may change to the second optional page “display setup” for the display settings of measuring graphs. Refer to Fig. 4-2.



Set the displayed range of Absorbance.

Maximum: 0.100

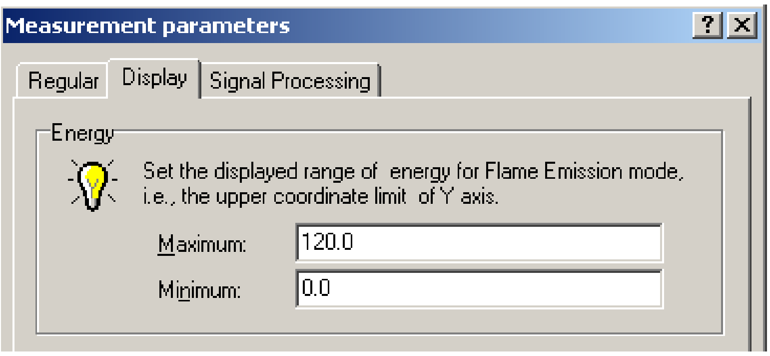
Minimum: -1.000

Set the interval time of signal profile update, during which signal profile will be continuously monitored.

Time: 10 Sec

Fig. 4-2 Page of setting display parameters

In this page, you can set the display boundary of absorbance in the measuring graphs, i.e. the lower and upper limits of the vertical / longitudinal axis of the measuring graphs. If you are selecting “Flame emission” as your current measurement mode, then you may also set the display range of energy. See also figure 4-3.



Set the displayed range of energy for Flame Emission mode, i.e., the upper coordinate limit of Y axis.

Maximum: 120.0

Minimum: 0.0

Fig. 4-3 Setting the energy range of flame emission analysis

“**Refresh time**” is the maximum time period during which the real time profile can continuously display in flame mode, i.e. the threshold of time axis (the horizontal or abscissa) in the measuring graph. For example, if you set the “Refresh time”as 10 seconds, then the maximum display period of time coordinate axis will be 10 seconds during the measurement, and the real time profile will be refreshed/renewed every 10 seconds, the former curves will be cleared out. Nevertheless, you need not to worry too much about that, since you may adjust the display range of your horizontal coordinate to a proper extent through the “graph display setup” function, i.e. you may display your former graph for the second time. The graph will be removed when you begin to make a second measurement. Note that the “Refresh time” can only be active in **Flame** mode. When you are using **Furnace** mode for making measurement parameter settings, it will not be displayed.

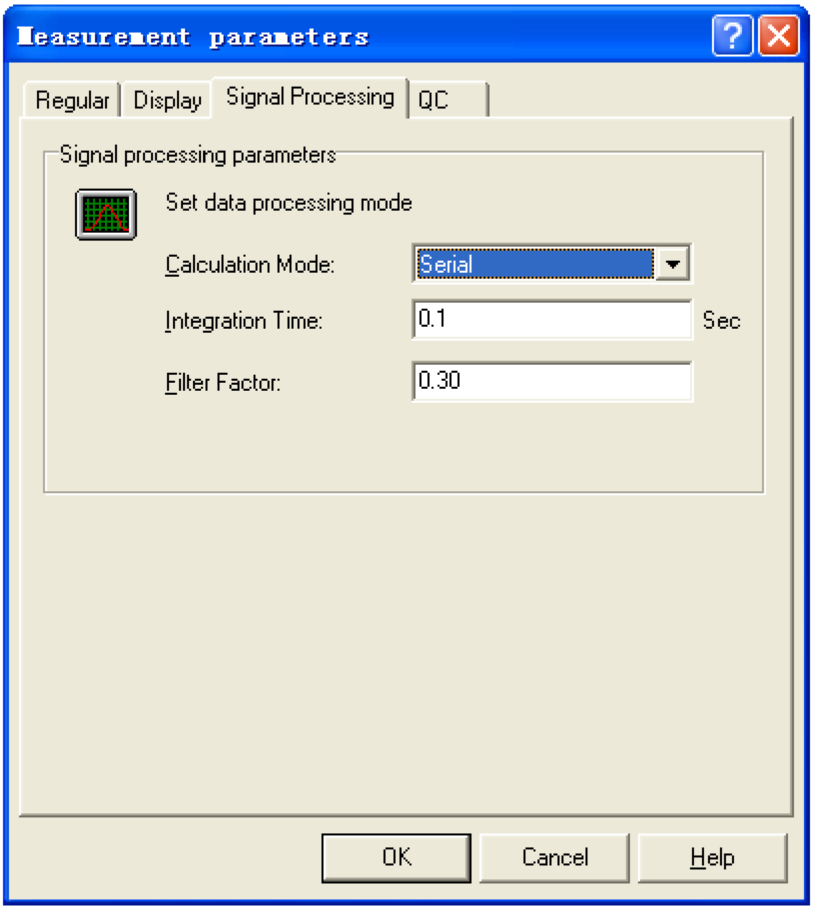
4.1.3 Signal processing

After making display setup, you can select the third page “Data processing” for signal processing parameter settings (See figure 6-4). In this page, you can check the data calculation mode from the “Calculation mode” pull-down menu box. The calculation mode you may select is as follows: continuous, peak height, and peak area. However, you can only choose two of them: **peak height** or **peak area** for the selection of calculation mode in furnace analysis.

You need also to input the integration time for the measuring data in the “Integration time” box. The integration time you enter should be at the range of 0.1 to 100 seconds. In Furnace analysis, the “Integration time” box will become gray (inactive). Customers need not to set the integration time in this mode, system will automatically take the total sum of the heating time in the atomization stage and the last three seconds in the former step (Ashing stage) in the furnace temperature program as the default integration time.

“Range extend” is the extending times of the received data. It should be controlled at the range of 0.01~100 times.

“Filter factor”is a constant which needs to be given to filter the acquired data, i.e. the flatten factor. It should be controlled at the range of 0.01~10. For the graphite furnace analysis, the factor sets 0.1.



Calculation Mode: Serial

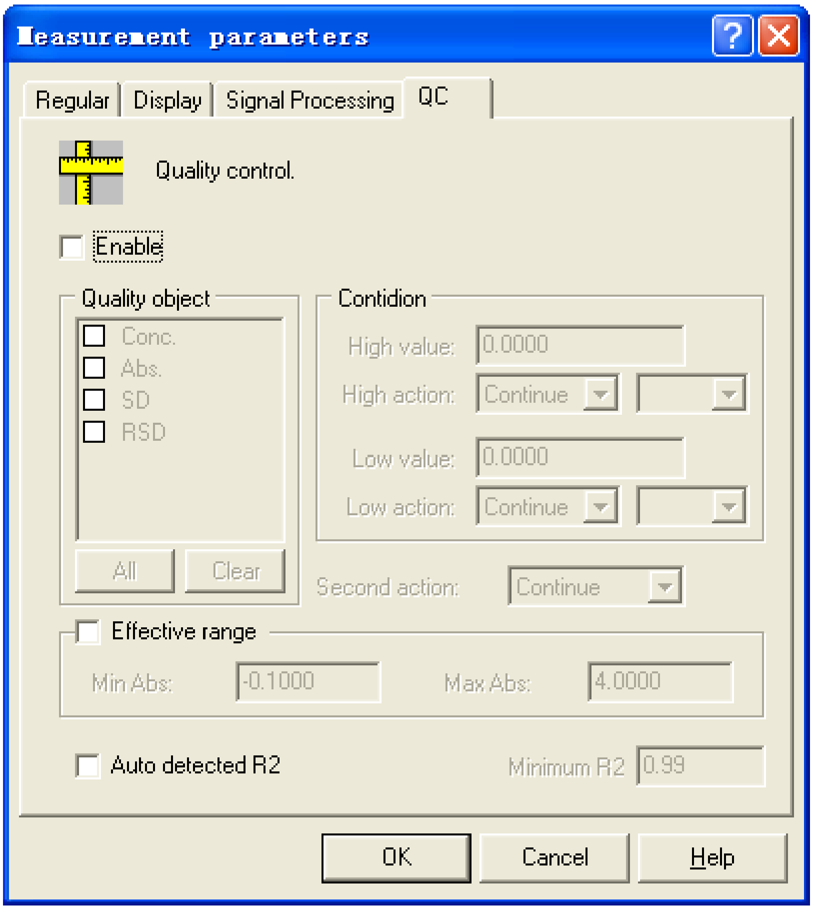
Integration Time: 0.1 Sec

Filter Factor: 0.30

Fig. 4-4 Signal processing

4.1.4 Quality control

Quality control is added to the software in order to cooperate with the auto-sampler. The user can monitor the measure results by using this function, and also can set control options by using this function. See Fig. 4-5.



Measurement parameters

Quality control.

☐ Enable

Quality object

☐ Conc.
☐ Abs.
☐ SD
☐ RSD

Contidion

High value: 0.0000
High action: Continue
Low value: 0.0000
Low action: Continue

Second action: Continue

☐ Effective range

Min Abs: -0.1000
Max Abs: 4.0000

☐ Auto detected R2
Minimum R2 0.99

Fig. 4-5 Quality control

- Upper value and lower value：

  Set the upper value and the lower value of the quality control.

- Deal with ultra-upper value and ultra- lower value:

  Set the processing method when the measure result is ultra-upper value. The user can choose “continue”, “stop”, or “re-measure”. In addition, the user can choose the marking, which will be marked in the ultra-upper value grid of the measure table, for the ultra-upper value.

- Deal with out of range for the second time:

  Set the processing method when the measure result is out of range for the second time. The user can choose “stop” or “continue”.

Control range of Abs: the user can choose effective monitoring range for Abs. Quality control can only monitor the data when the Abs is in the range.

4.2 Setting measurement samples

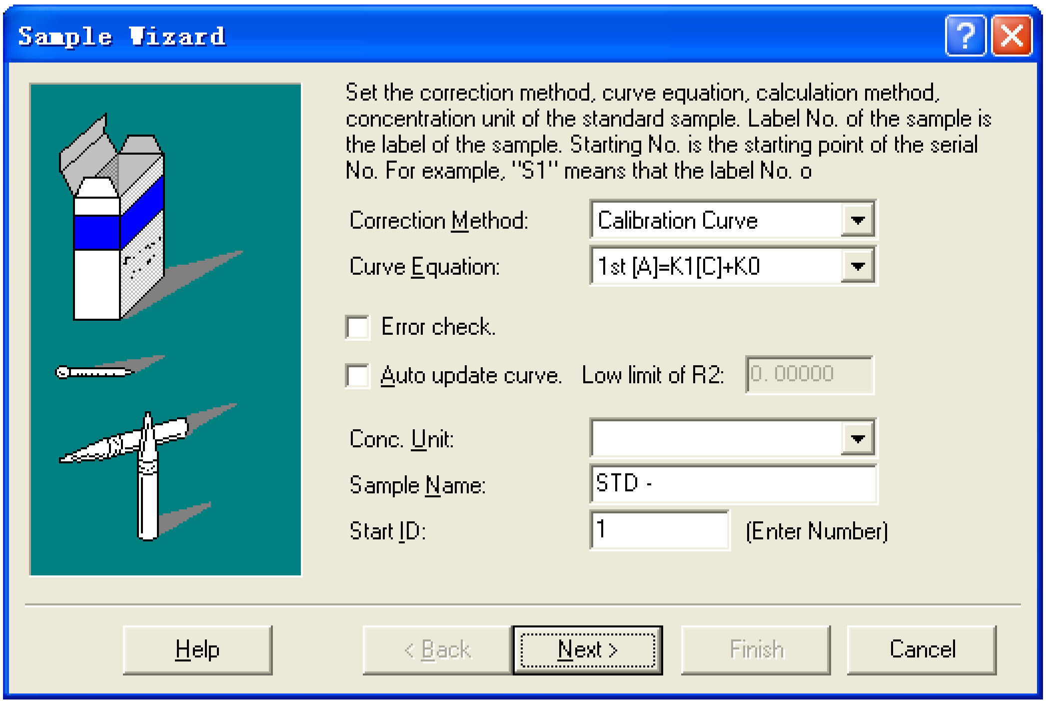
Before making a measurement, you need also to set the measuring samples. Select [settings]—[Guide to the sample setup] from the menu or just press the button “” from the toolbar to open the “Guide to the sample setup” dialog box. It includes three pages allowing you to make measurement sample settings step by step. The first page is meant for standard sample settings referring to Figure 4-6.

“Correction method” is used to select the correction method for standard samples. The method to be selected is as follows: calibration curve, standard addition or interpolate method.

“Regression equation” is the calculation formula selected for the regression of calibration curve of the standard samples, including “linear $A=K_1[C]+K_0$”, “linear $C=K_1[A]+K_0$”, “square $C=K_2[A]^2+K_1[A]+K_0$” and “cubic $C=K_3[A]^3+K_2[A]^2+K_1[A]+K_0$” etc. This section allows you to select a proper regression order for the calibration curve.

“Concentration unit” is the unit used for the sample concentration. It may be as follows: “ng/µl”, “ng/ml”, “µg/µl”, “µg/ml”, “mg/l”, “ppb”, “ppm”, “mol/l” etc. You can also enter the unit by yourself in the case of no proper unit existing in the concentration unit list.

“Sample identification (ID)” and ”Serial number” generally make up the sample number. For example, Sample no. “S1” stands for the sample which has a Sample ID ”S” and a Serial number “1”.



Set the correction method, curve equation, calculation method, concentration unit of the standard sample. Label No. of the sample is the label of the sample. Starting No. is the starting point of the serial No. For example, "S1" means that the label No. o

Correction Method: Calibration Curve

Curve Equation: 1st [A]=K1[C]+K0

☐ Error check.

☐ Auto update curve. Low limit of R2: 0. 00000

Conc. Unit:

Sample Name: STD -

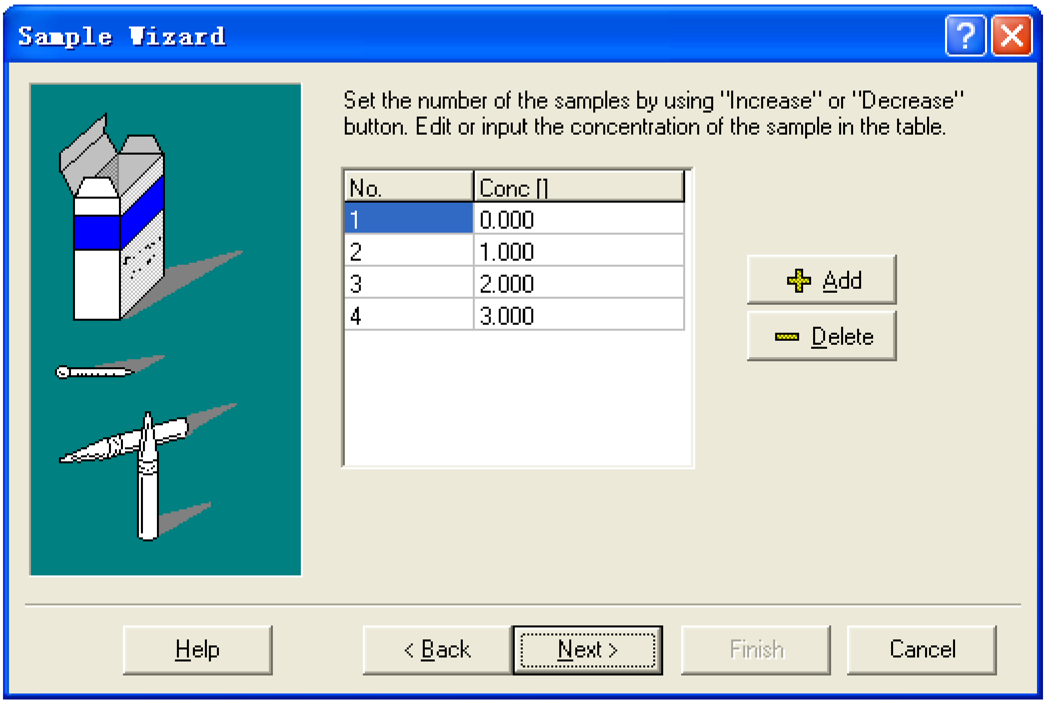
Start ID: 1 (Enter Number)

Fig. 4-6 Standard sample setting

“start error correction”: It can effectively reduce the measure error. If your measure result is not accurate, you can choose this option.

“Auto-upgrade equation”: It can monitor the standard equation according to the minimum relativity you set. Once the relativity is less than the minimum relativity you set, the system will upgrade the standard equation from linear equation to nonlinear equation. And calculate again. The function is effective only when the curvilinear equation is first order equation.

After you have made standard parameter settings, click the “Next step” button entering the page for standard concentration setup referring to figure 4-6. You can enter the concentration values in the concentration boxes of the standard list. Click “Increase” button to add the standard samples, you may add totally 8 samples in the standard list. Click “Decrease” to remove/delete standard samples, you should leave at least one standard sample in the list.



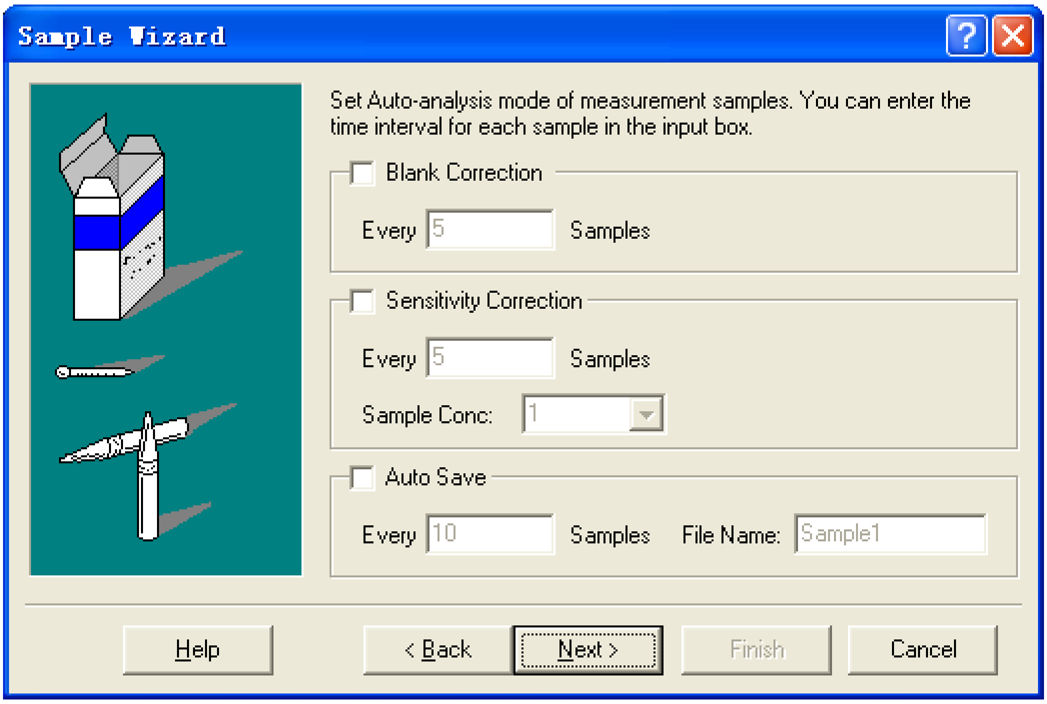
Sample Wizard

Set the number of the samples by using "Increase" or "Decrease" button. Edit or input the concentration of the sample in the table.

No.	Conc []
1	0.000
2	1.000
3	2.000
4	3.000

Fig. 4 -7 Setting the concentration of standard samples

After finishing the standard concentration setup, click the “Next step” button for the auto-analysis setup referring to figure 4-7. In this page, you may check some auto-analysis functions and make the corresponding parameter settings for the measurement.



Sample Wizard

Set Auto-analysis mode of measurement samples. You can enter the time interval for each sample in the input box.

☐ Blank Correction
Every 5 Samples

☐ Sensitivity Correction
Every 5 Samples
Sample Conc: 1

☐ Auto Save
Every 10 Samples File Name: Sample1

Fig. 4-8 Auto-analysis setup

- Blank correction:

  Blank correction is measuring the blank samples before measurement. Then the system calculates other samples according to the measure results.

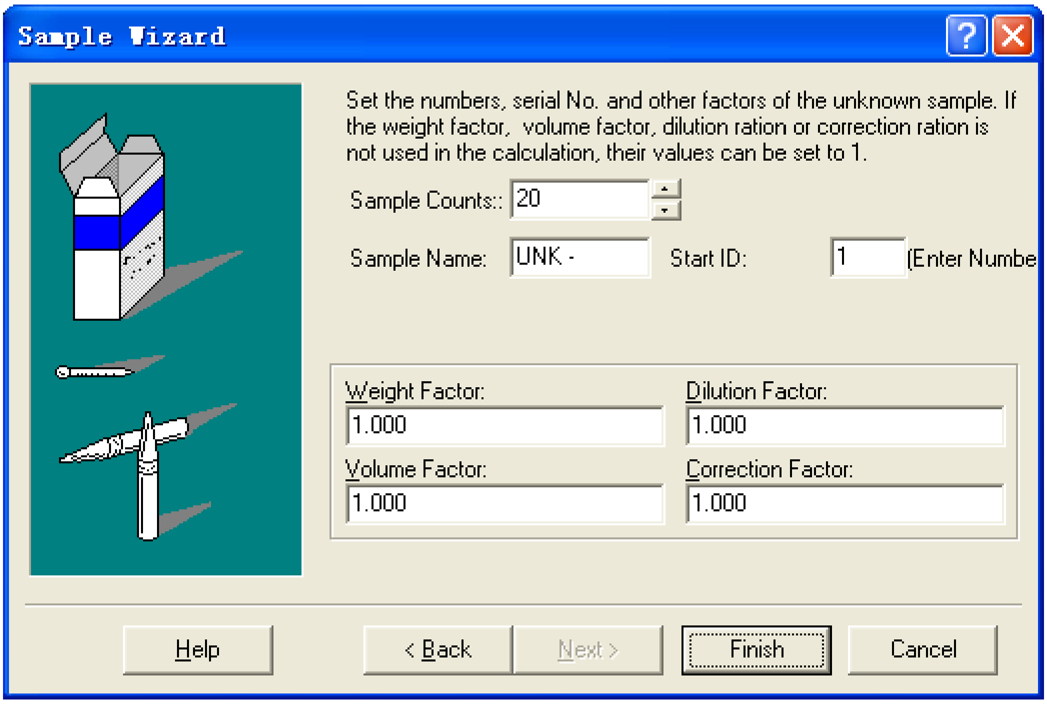
- Sensitivity correction:

  There may be some errors exist in your instrument for some reasons, called “drift”. Sensitivity correction function is added in the software in order to reduce errors. It needs to measure the standard sample, and then correct the sensitivity according to the measurement result. The user needs to select the serial number of the standard sample and correction cycle.

- Auto-save:

  Set the sample interval for storing measurement data in case that the measurement data will not be totally lost when the computer is deadly locked by accident. You can set a cycle, then the system will auto save your sample by your set cycle.

After you complete the auto-analysis setup, click the “Next setup” button entering the last page of this sample setup guide — the “Unknown sample settings” section, see figure 4-9.



Sample Wizard

Set the numbers, serial No. and other factors of the unknown sample. If the weight factor, volume factor, dilution ration or correction ration is not used in the calculation, their values can be set to 1.

Sample Counts:: 20

Sample Name: UNK -

Start ID: 1

(Enter Numbe

Weight Factor: 1.000

Dilution Factor: 1.000

Volume Factor: 1.000

Correction Factor: 1.000

Fig. 4-9 Setting the unknown samples

In this page, the following items will be set: number of unknown samples, sample ID and a variety of coefficients or factors needed for concentration calculation or correction. In the measuring result table, one column entitled ” Actual concentration” is listed. Its calculation formula is as follows:

$$\text{Real Conc.}=\frac{\text{Conc*Volume Factor*Dilution Ratio*Correction Factor* Sensitivity Correction Factor}}{\text{Weight Factor}}$$

You can input factor according to the sample, and then calculate the concentration. When you start the sample setup guide, the default factor is 1. That is to say, if you don’t make a setup, the “real concentration” is equal to “concentration” in the sample table after measurement. If you need to modify the above factor, you can use the edit function of the measurement table.

After you finish all the above sample parameter settings, you can make some changes through clicking the “former step” button and editing the contents of the former pages. When you confirm that you have properly made all the sample parameter settings and click the “Finish” button, system will transform your settings into a measurement table. Now you may start to make a measurement.

4.3 Start analysis

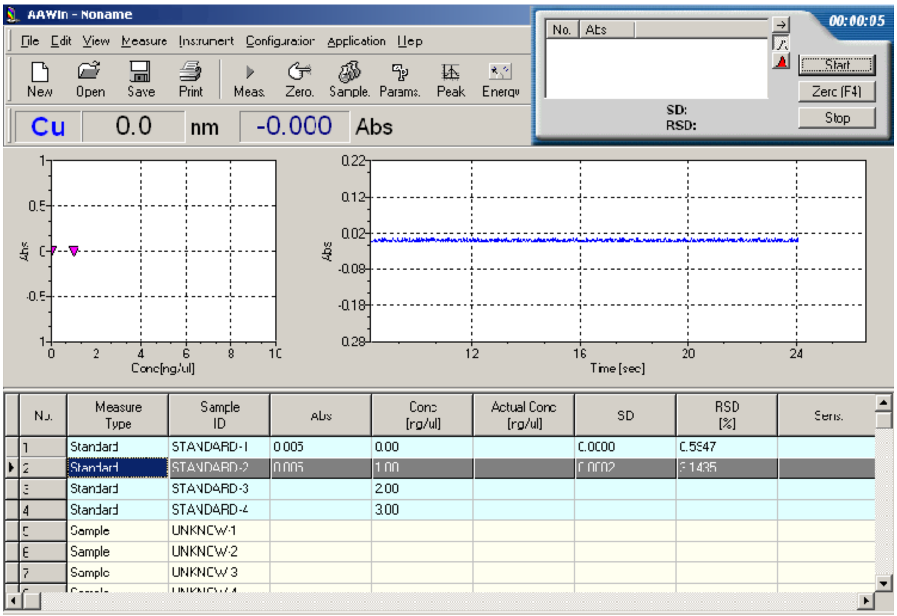
In AAWin software system, different analytical method needs to take different procedure and require different preparations to carry it out. In the following we will introduce you both the analytical procedure for Flame method and that for Furnace method. Please read the related contents carefully in case that any safety problem occurs.

4.3.1 Flame analysis

Note: Insert the baffle between the flame burner head and the graphite furnace before ignition. In addition, as to the manual exchange instrument, the exchange platform must exchange to the flame mode, then insert the baffle, and at last ignite.

In the case of flame analysis, you should carefully check all the piping tubes and the water seal for gas leak before making a measurement. When you make sure that everything is O.K, select [Instrument]—[Ignition] from the menu or click the “⚡” button from the toolbar for igniting the flame. When the flame height is too high or too low or if the burner is not set in proper position, turn to the “Burner parameter settings” for adjustment (See Section 3.8).

After you finish flame igniting and make proper adjustment to the burner parameters, you may now start to make a measurement. To do so, first select [Measurement]-[Start] from the menu or click the button“▶”from the toolbar or just press F5 key to open the measurement window, then there will be analog signal coming up in the real time graph. Refer to Figure 4-10.

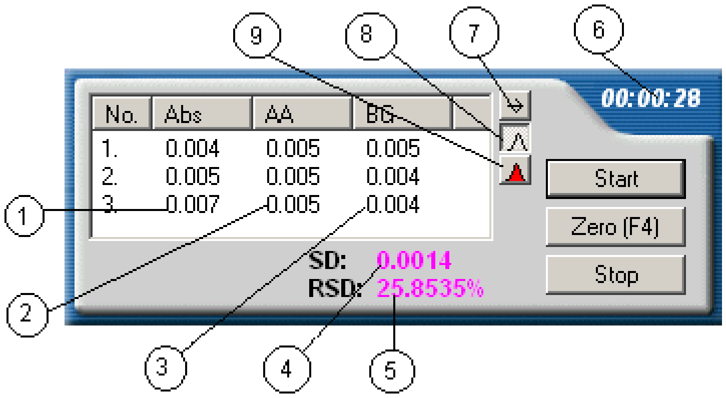


No.	Measure Type	Sample ID	Abs	Conc [ng/ul]	Actual Conc [ng/ul]	SD	RSD [%]	Sens.
1	Standard	STANDARD-1	0.005	0.00		0.0000	0.5947	
2	Standard	STANDARD-2	0.005	1.00		0.0002	3.1435	
3	Standard	STANDARD-3		2.00				
4	Standard	STANDARD-4		3.00				
5	Sample	UNKNOW-1						
6	Sample	UNKNOW-2						
7	Sample	UNKNOW 3						

Fig. 4-10 Measuring window

Now you can click the [Start] button for sampling. If you have chosen a Manual mode to make measurement in the measurement parameter-setting window, you will have to push the [Start] button again for another measurement after you finish every single sampling operation. In the case of Automatic mode, however, system will automatically take the sampling process according to your setting of the “Time interval”. After finishing the samplings, system will display the results in the measurement window, standard deviation (SD) and relative standard deviation (RSD) values will also be calculated and displayed in the window after the third repetitive sampling. After you complete all the samplings set by the duplicate number in the measurement parameter dialog box, system will calculate the mean value of the measurement results and display it in the proper cell (unit element) of the measurement table. See also Figure 4-10.

During measurement a timer is placed on the upper right of the measurement window, from which you can check the total time used for your analysis. You can also insert a blank before each sampling for zero check. Click the “Zeroing” button to make zero adjustment. Press the “End” button when finished the measurement, see also figure 4-11. If you have selected D2 lamp or SR mode for the background correction, AA and BG values will be displayed together with the absorbance (net value) in the measurement window. In the mean time, the AA and BG curves will also appear in the real time graph. In this case, the absorbance is the difference subtracting the BG value from the AA value (Abs. = AA — BG). If no background correction is used, system will display the absorbance and provide the Abs. Curve only. In this situation, the abs. is equal to the AA value (Abs. = AA). Curve color in the real time graph can be set through the function of the Graph Setup. In the case of default, Abs. Curve takes blue, AA curve is light green and BG curve for red.



No.	Abs	AA	BG
1.	0.004	0.005	0.005
2.	0.005	0.005	0.004
3.	0.007	0.005	0.004

SD: 0.0014
RSD: 25.8535%

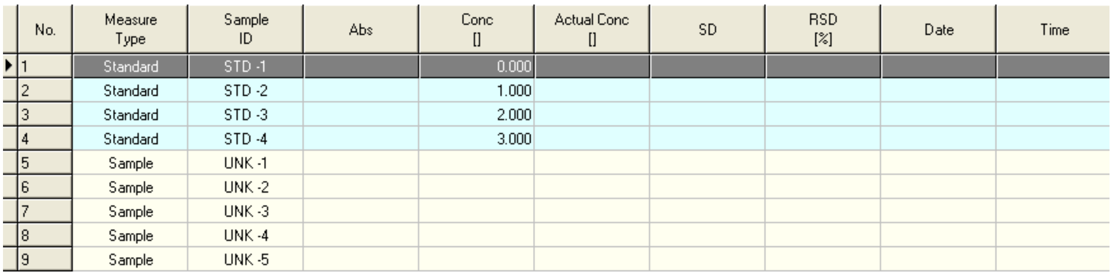
00:00:28

Fig.4 –11 Measurement for flame analysis

① Absorbance(Net value)
② Atomic absorption value(AA value)
③ Background value(BG value)
④ Standard deviation
⑤ Relative standard deviation
⑥ Measurement timer
⑦ Results for continuous integration mode
⑧ Results for peak height calculation mode
⑨ Results for peak area calculation mode

Use the ⑦ ⑧ and ⑨ three buttons for the changeover of the calculation mode between continuous, peak height and peak area.

In AAWin software system, measurements are made sequentially according to the measurement table. After you finish your current sample, click “Start” from the measurement window for the next sample. The current measuring sample in the measurement table will be automatically changed to the next. If you are measuring the last sample as for the current sample, system will add a new unknown sample in the measurement table for continue when you press the “Start” button.



No.	Measure Type	Sample ID	Abs	Conc []	Actual Conc []	SD	RSD [%]	Date	Time
1	Standard	STD -1		0.000					
2	Standard	STD -2		1.000					
3	Standard	STD -3		2.000					
4	Standard	STD -4		3.000					
5	Sample	UNK -1							
6	Sample	UNK -2							
7	Sample	UNK -3							
8	Sample	UNK -4							
9	Sample	UNK -5							

Fig. 4-12 Displaying the auto-filled measured results

In the case of calibration curve preparation, system will draw up each point of the standard samples on the graph of the calibration curve after sampling, and display the final calibration curve together with the regression calculation results when all the standards you have set in the measurement table are finished. See Fig. 4-10.

Next, you can measure the unknown samples. The measurement results will be auto-filled into the measurement table. When you finish all the measurement of samples, you may close the measurement window. If you need to save your measurement results, select [File]—[Save] from the menu or click the toolbar “💾”。 When you want to print them out, select [File] —[Data Print] from the menu or click the toolbar “🖨” for output, then choose “Form” in the pop-up window as for data source confirm.

By now, we have introduced you the whole measure procedure for the flame method. In the following, we will present to you the measure steps for the furnace method.

4.3.2 Graphite furnace analysis

As furnace method can provide sample analysis with much higher sensitivity than flame method, please turn to the furnace mehod when you need to make measurement with trace or super trace level, where high sensitivity is preferred for analysis. Different from flame method, furnace analysis donnot need to make an ignition. But you have to make more delicate connections for gas piping tubes or water hoses. Before measurement, please carefully check all the piping tubes for leak. If you are making the first measurement for furnace analysis after you switch on the power of the instrument, you need to set the heat program first. When you make sure that you have already properly set the temperature program, select [Measurement]—[Start] from the menu or click the toolbar “▶” or just press F5 key. System will change to the measurement window.

For furnace analysis, there will not be signal profile displayed in the measurement window as will do for flame method before you click the [Start] button from the window. For the first you need to properly load your sample into the graphite tube, you can then click “Start” for running the furnace temperature program. In this time, signals begin to appear in the measurement window, and the current furnace temperature, heat stage together with a timer, which will indicate the time left for the heat stage, will also be displayed in the window. Refer to Figure 4-13.

00:00:12

No.	Abs	AA	BG
1.	0.002	0.000	0.003
2.	0.011	0.005	0.002
3.	0.009	0.005	0.003

Step 4 of 4 SD: 0.0047

(0) 0 'C RSD: 63.6404%

Fig. 4-13 Measurement for graphite furnace analysis

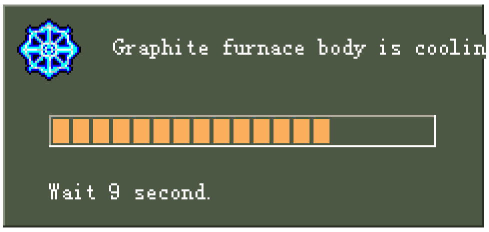
①The current heat step

②Number of the total heat steps

③Timer for how much time is left for the current heat step

④The current temperature

After each single measurement, system will pop up a message box indicating that the furnace body is cooling down and showing through a timer how much time will be still left until the next sample injection can be performed. During this period, you cannot make any other operations through the software. You can make a further measurement only when the timer is over. See Figure 4-14.

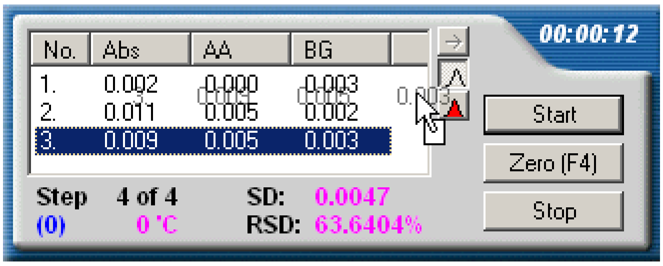


Graphite furnace body is coolin

Wait 9 second.

Fig. 4-14 Cooling down the furnace body

Like in the flame method, standard deviation (SD) and relative standard deviation (RSD) values will also be displayed after the third measurement (It is so called sampling in flame method) in furnace analysis. As there may be a variety of factors including the status of the instrument affecting the repeatability of the measurement results during furnace process, some measure results may be unacceptable. In this case, you can measure the sample for one extra time. You can just reload the sample, click with the left key of the mouse on the last measure result and drag it onto the [Start] button and then release the mouse to make a repetitive measurement of the sample. The former result will then be overwritten by the newly measured result, the standard deviation (SD) and relative standard deviation (RSD) values will also be re-calculated, refer to Figure 4-15. Note that only when you have performed measurement of the same sample for above 3 times (inclusive) can you make a repetitive measurement. This can be also applied to the flame method. However, as flame method can generally give much better repeatability than furnace method, this function may not be so commonly used especially in the "Automatic" mode.



00:00:12

No.	Abs	AA	BG
1.	0.002	0.000	0.003
2.	0.011	0.005	0.002
3.	0.009	0.005	0.003

Step 4 of 4 SD: 0.0047

(0) 0 'C RSD: 63.6404%

Fig. 4-15 Renewing measurement for the measured result

As furnace analysis may take you much longer time for measurement, you are strongly recommended to follow the procedure exactly. After you complete your measurements, you can save your measure results or print them out. Other measure steps are similar to that of the flame method thereby will here not be involved.

4.3.3 Renew analysis

The “Renew analysis” function, which will be here introduced, is different from what we called the “Re-measurement” in the above section. Re-measurement can be defined by the replacement or update of the individual results for the duplicate measurements of one sample. The re-measurement results will be taken for the calculation of the final result of the sample. Renew analysis, however, can be regarded as the re-determination of the sample, which has already been measured for a final result, i.e. the replacement or update of the final result.

If you find that some measure results are unacceptable or need to be further improved after you have finished all the measure samples, turn to the renew analysis function of AAWin software. To do so, you first highlight the sample in the measurement table with the mouse, then select [Measurement]—[Renew analysis] from the menu or right click on the measurement table and select [Renew analysis] from the pop-up menu to make the renew analysis. After measurement, you may also leave the window open and click [Start] again to make the renew analysis for a second time if you are not satisfied with your measure result. You can keep doing so again and again until the measure result is acceptable.

When you are satisfied with your renew analysis result, click the [End] button for closing the measurement window. If you continue making renew analysis of other samples, click the toolbar button “▶” after you quit from the last renew analysis window.

While you make renew analysis of standard samples, the calibration curve will be recalculated and refitted according to your renew analysis results.

4.4 Shut down the AAWin system

When you have finished all the measurements and made a print-out of the measure results or saved them to the disk, you can select [File]—[Exit] from the menu to quit from the AAWin software system. Before closing AAWin system, a message box will pop up to remind you to follow the proper procedure to shut down the instrumental system. This warning message will always come up every time you close the AAWin software system in case that you may forget to shut down the water, gas or electricity supply or take some safety precaution measures after you complete your measurement. Therefore, you are assumed to follow exactly the shut-down procedure to quit from the analysis, i.e. after finishing measurement and closing the AAWin software, switch off the water, gas and power supply immediately in case of any accident.

4.5 Summary

By now we have introduced you the whole process of both flame and furnace methods for atomic absorption spectrophotometer, you are sure to know the AAwin system very well. Nevertheless, you need also to get more practice to enhance your operational capacity of the instrument.

Chapter 5 Application

Through the reading of the former chapters, you are sure to have made a better understanding of the AAWin operation system for AA-990 series atomic absorption spectrophotometer. In the next, we will introduce you a full measure procedure through an example.

5.1 Preparation for analysis

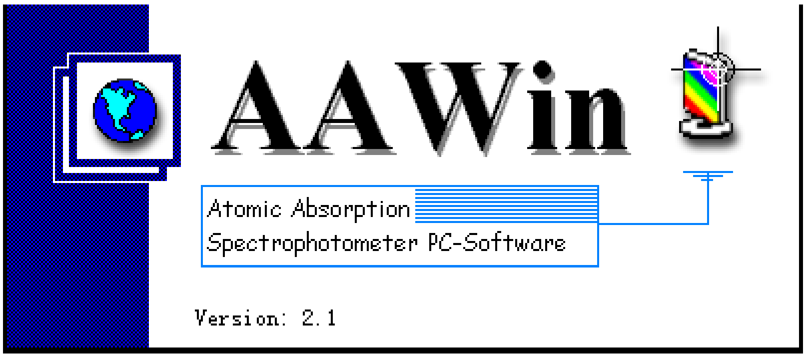
In the first, you should define the elements that need to be measured in the unknown samples. We will here take the measure of the Cu element in the sample as an example to provide you the full measure procedure of the AA analysis. Prepare the Cu element lamp, Cu standard solutions, blank and the unknown sample solutions as well. Check if the pressures of the acetylene gas cylinder and/ or the argon gas cylinder are enough for use. Before making a measurement, you should get all these preparation works ready.

5.2 Operation procedures

When the preparation work got ready, you may follow the following procedures to perform the instrumental operations.

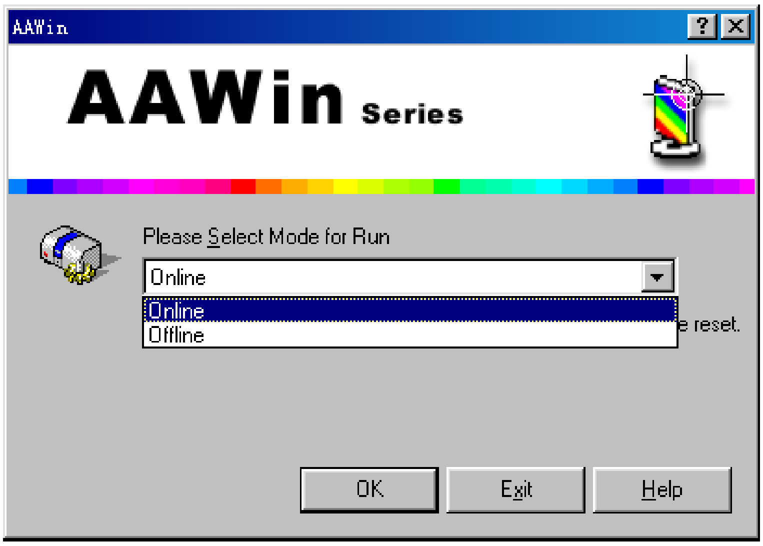
Step 1: Instrumental initialization

When you start up AAWin software, switch on the power of the AA computer and run WINDOWS operating system first, and then double click the AAWin software icon on the desktop or click the AAWin entry under the group of "**AAWin for AA-990**" from the program group. System will run the AAWin software, and a starting headline window as below will pop up on the computer screen.

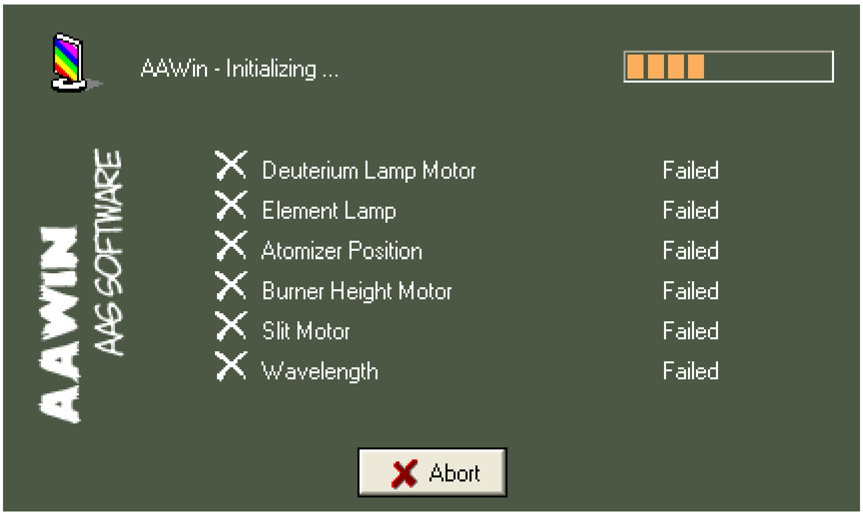


This starting window will soon disappear when you turn on the power for the AA instrument and properly connect the communication cable. If it is not the case, a message box will soon pop up reminding you to check the connections. When you make sure that all the connections are in normal, click "**Retry**" for re-communication with the AA main unit. The starting window goes away after the communication with the AA main unit is successfully accessed.

A run mode selection box will come up next for you to choose a proper run mode of the software shown as below. Select "**Online**" from the dropdown box for a run mode selection and click "**Determine**" to enter into the instrumental initialization.



Please Select Mode for Run: Online



AAWin - Initializing ...

✗ Deuterium Lamp Motor	Failed
✗ Element Lamp	Failed
✗ Atomizer Position	Failed
✗ Burner Height Motor	Failed
✗ Slit Motor	Failed
✗ Wavelength	Failed

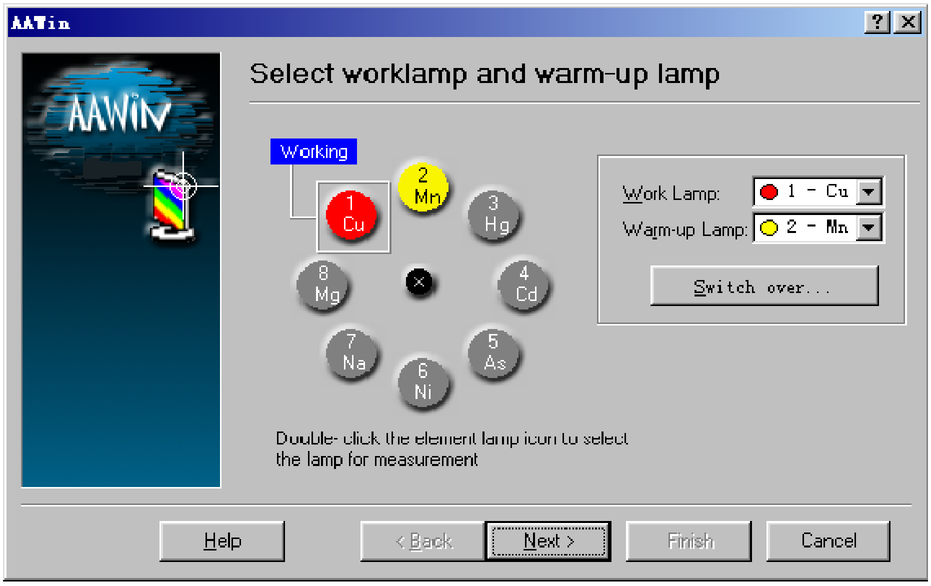
The instrumental initialization is mainly aimed at the setup of initial parameters of the individual drive motors including D_2 lamp motor, HCL lamp motor, atomizer motor, burner motor, slit width motor and wavelength motor as well. When a certain entry is successfully initialized, a check mark ✓ will come up on the left beside the item; Otherwise, a cross sign ✗ will turn up. If any one of them fails to be success fully initialized, the whole instrumental initialization will not be passed. In this case, a message box will come up after the initialization for you to choose whether you want to try it again or not. You may enter "**Yes**" to continue or press "**No**" to exit the AAWin system.

Note: When you want to run the AAwin software for a second time after you exit the software without the cutoff of the AA main unit power, you can start it up without a further instrumental initialization, supposed that you have already performed the instrumental initialization successfully in the first time. In this case, you can select the "Offline" mode from the pull down box for the run mode selection and click "Determine" to skip the initialization

procedure and directly enter into the operation system. Next you select “Online” mode from the [Application] menu to make communication with the AA instrument. On the contrary, you can also disconnect the AAWin software from the AA main unit when you perform in the online mode. If the current mode is online, there will be a check mark “ ✔ ” coming up beside the “online” entry, otherwise, the check mark will not appear. In addition, the specified element lamp needs to be normally lit up during the instrumental initialization, and the light beam from the element lamp should pass through the atomizer smoothly. Otherwise, the wavelength motor initialization will fail. In that case, you can’t perform the line search and therefore can’t make measurement either.

Step 2: Set the element lamp

When you finish the instrumental initialization, system will pop up the element lamp selection window, which will take the form of a setup guide, allowing you to make lamp and its element parameter settings stepwise. The guide contains totally four pages, in the first page you are supposed to set the working lamp and the warm-up lamp referring to the Figure as below. You may also select a working or warm-up lamp from their individual dropdown boxes, or you can make exchange of them through the exchange button.



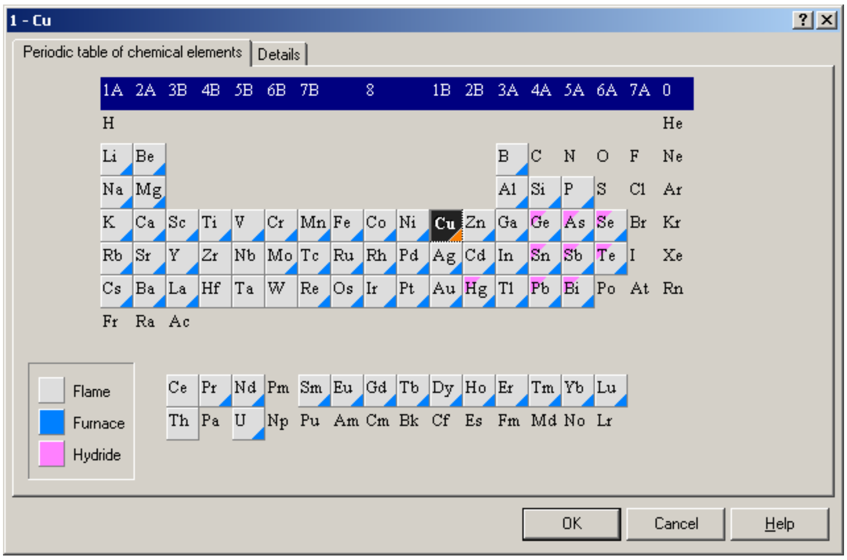
Select worklamp and warm-up lamp

Work Lamp: 1 - Cu

Warm-up Lamp: 2 - Mn

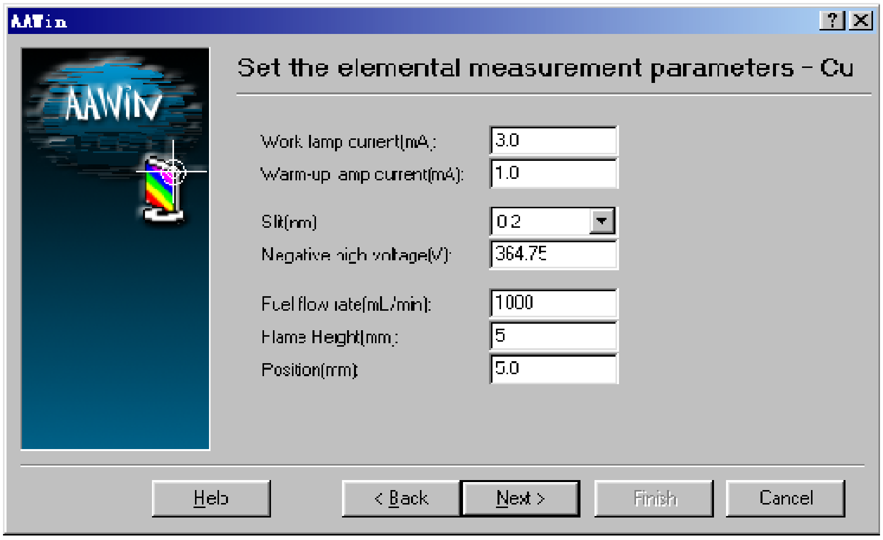
Double- click the element lamp icon to select the lamp for measurement

You can load cupper HCL lamp in the No.1 lamp turret and set it as the working lamp, choose another turret number for the next element to be measured as the warm-up lamp, then click “**Next step**” to set the element lamp parameters. If there is no Cu lamp mark coming up in the lamp turrets, you may select one turret number (e.g. No.1) and unplug the element lamp in this turret then insert the Cu lamp. Move your mouse to the turret number position (No.1) and double click the turret icon entering into the element selection box, see figure as below.



Point the mouse to the Cu element position in the periodical table and make a double click or click the Cu element followed by pressing the **”determine”** button in the popup box to return to the element lamp selection window. You will see that Cu element has already set on the No.1 lamp turret. Afterwards, click “**Next step**” to switch to the element lamp parameter window.

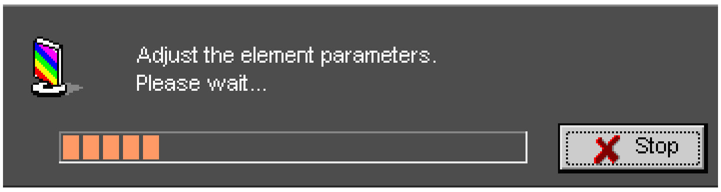
Step 3: Set the element lamp parameters



Set the elemental measurement parameters - Cu

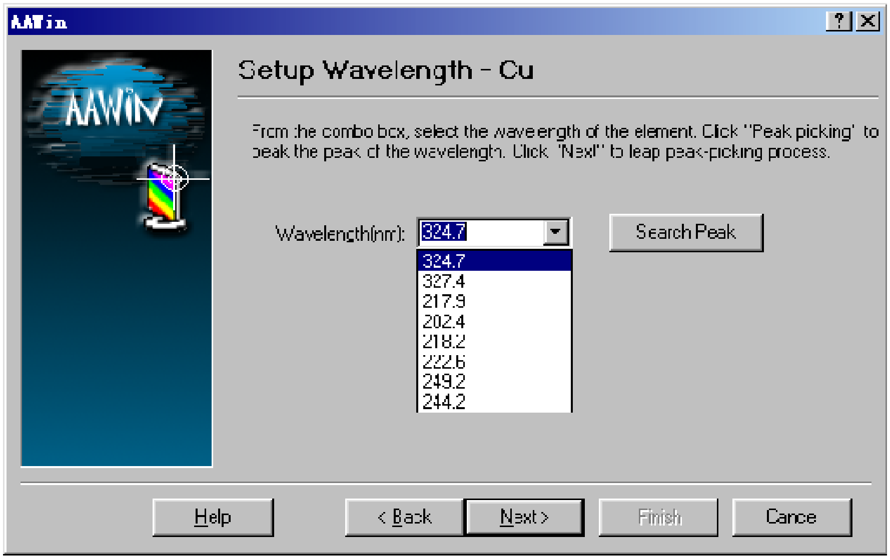
Work lamp current(mA): 3.0
Warm-up lamp current(mA): 1.0
Slit(nm): 0.2
Negative high voltage(V): 364.75
Fuel flow rate(mL/min): 1000
Flame Height(mm): 5
Position(mm): 5.0

The parameters shown as above can provide a reference when setting the instrumental parameters. You may set the instrument following the above values, or make a modification according to the requirement for the measurement. The parameters needed to be set are: the working lamp current, the warm-up lamp current, bandwidth and the negative high voltage. Click the “**Next step**”, system will send the above parameters you have set to the AA main unit through the instruction transfer window as follows:



Adjust the element parameters.
Please wait...

After you have finished the above settings, system will switch to the wavelength setup page (see figure below). In this page, you can choose the characteristic wavelength for the specified element and perform the peak search. Select a resonance line from the dropdown box of wavelength, and click the "**Peak-search**" button, system will perform the peak search for the specified wavelength.



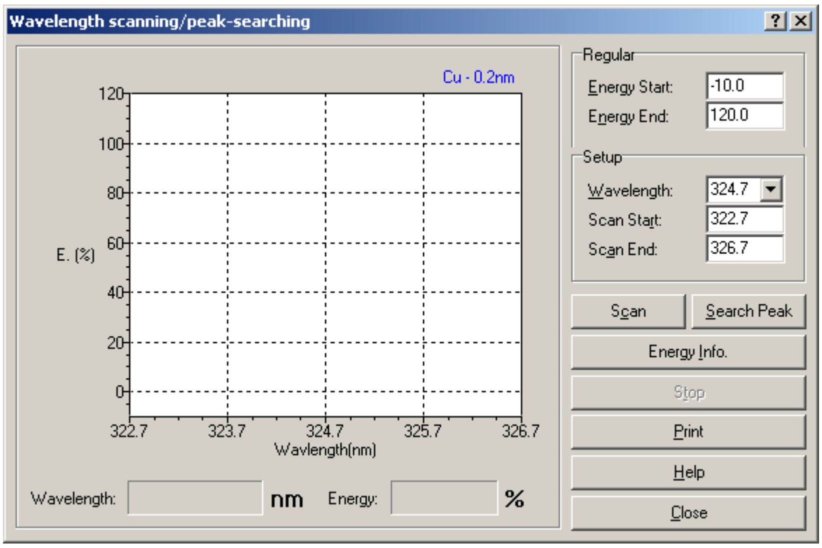
Setup Wavelength - Cu

From the combo box, select the wavelength of the element. Click "Peak picking" to peak the peak of the wavelength. Click "Next" to leap peak-picking process.

Wavelength(nm): 324.7

324.7
327.4
217.9
202.4
218.2
222.6
249.2
244.2

Window for the peak search of the specified wavelength is shown as below:

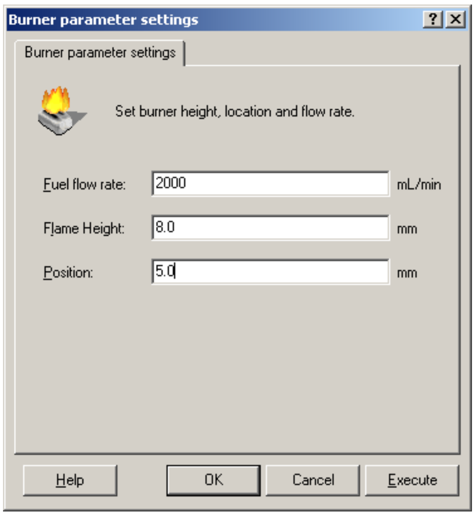


Energy Start: -10.0
Energy End: 120.0
Wavelength: 324.7
Scan Start: 322.7
Scan End: 326.7
Wavelength: nm Energy: %

When peak search is complete, click "**Close**" to quit from the wavelength setup window. Press the "**Finish**" button to end up the element lamp setup and exit the setup guide.

Step 4: Set the atomizer position

System will next change to the measure procedures. Select [**Setup**]—[**Set the burner parameters**] from the menu to open the burner setup box (see Figure). You can set the parameters as fuel gas flow rate, burner height and burn position etc in the burner setup box.



Set burner height, location and flow rate.

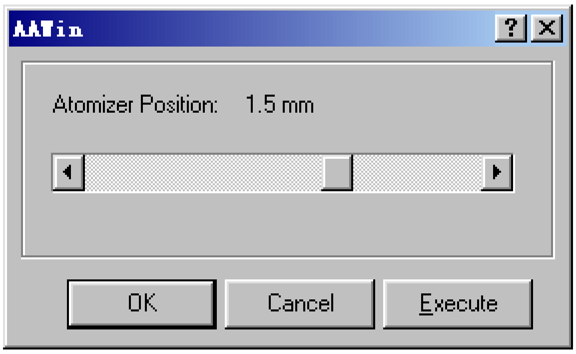
Fuel flow rate: 2000 mL/min

Flame Height: 8.0 mm

Position: 5.0 mm

Set the **Gas flow rate** to the value of 1200~1800mL/min. After ignition, you can make a readjustment to the **Gas flow rate** until you obtain the flame type of what you want (such as lean fuel flame, abundant/rich fuel flame and stoichiometric combustion flame etc.). Set the **Burner height** to the value of 4~10mm；Set the **Burner position** until the central axis of the element lamp light path is right located above the burner slit. You may take a piece of paper to observe the relative light spot position to the burner slit on both the left- and right- end of the burner head to make adjustment.

When you perform measurement in furnace mode, select [**Setup**]—[**Set the atomizer position**] from the menu after you have load the graphite furnace (see figure as below).

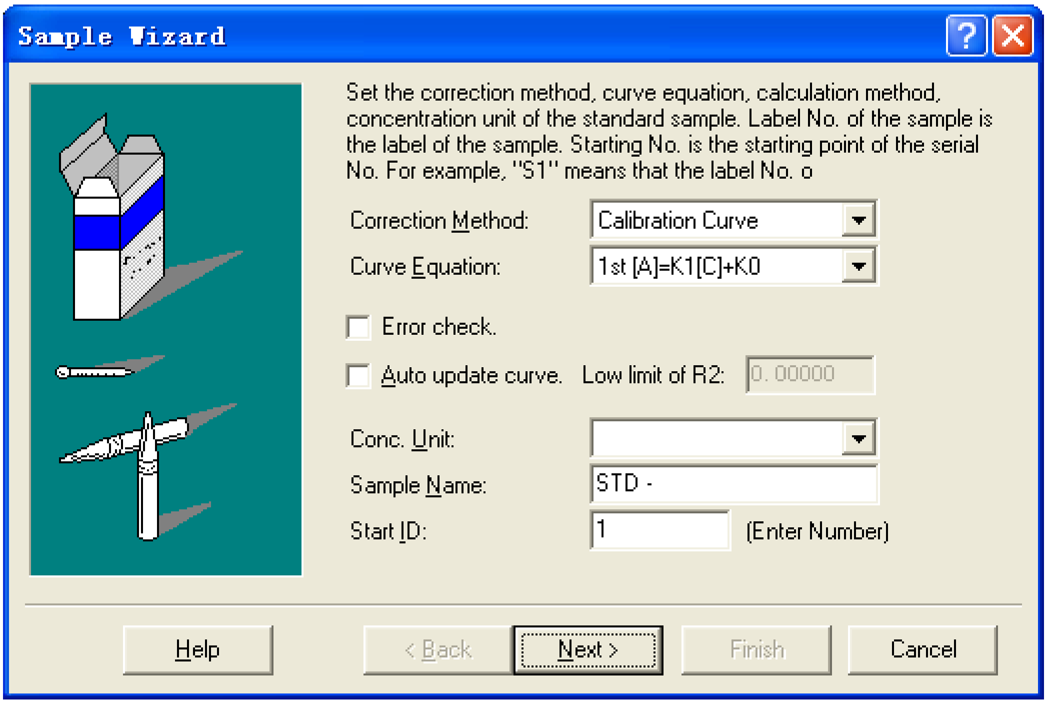


Atomizer Position: 1.5 mm

Use the left mouse key to drag the scroll bar back and forth for adjustment. After you have set a position for the scroll bar, click [**Determine**] to activate your setup. Check the instrumental energy state until it shows the maximum value. If the energy exceeds 120%，lower the instrument energy to 100% and then make adjustment to the atomizer position until the energy indicates a maximum value.

Step 5: Set the samples

Before performing measurement, you need to set the measure samples first. Select [**settings**]—[**Guide to the sample setup**] from the menu or press the button “” from the toolbar to open the “**Guide to the sample setup**” dialog box. The sample setup guide includes three pages in all allowing you to make measure sample settings step by step. The first page is used for standard sample settings referring to the figure as follows:



Set the correction method, curve equation, calculation method, concentration unit of the standard sample. Label No. of the sample is the label of the sample. Starting No. is the starting point of the serial No. For example, "S1" means that the label No. o

Correction Method: Calibration Curve

Curve Equation: 1st [A]=K1[C]+K0

☐ Error check.

☐ Auto update curve. Low limit of R2: 0. 00000

Conc. Unit:

Sample Name: STD -

Start ID: 1 (Enter Number)

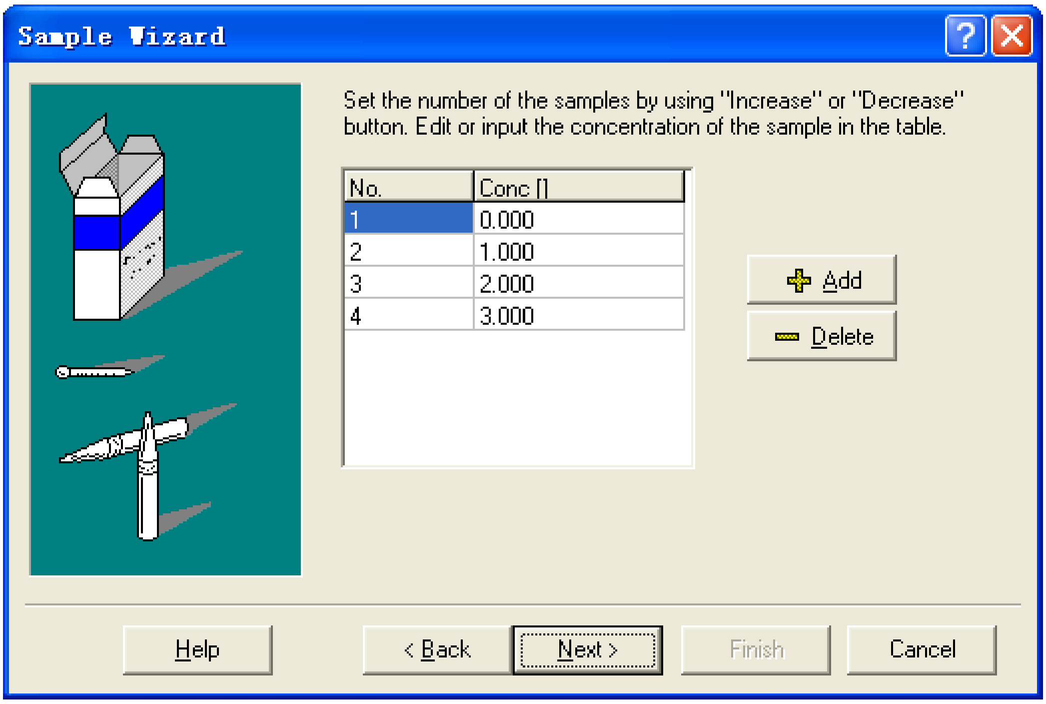
"Correction method" is used to set the correction method for the standard samples. The methods that are available are given as follows: **calibration curve**, **standard addition** and **interpolate method**.

"Regression equation" is the calculation formula selected for the regression of calibration curve of the standard samples, including "linear $A=K_1[C]+K_0$", "linear $C=K_1[A]+K_0$", "square $C=K_2[A]^2+K_1[A]+K_0$" and "cubic $C=K_3[A]^3+K_2[A]^2+K_1[A]+K_0$" etc. This section allows you to select a proper regression order for the calibration curve. Usually, a linear (first-order) regression of the calibration curve is used for calculation of the measure results.

"Concentration unit" is the unit used for the sample concentration. The units that can be used are as follows: "ng/μl", "ng/ml", "μg/μl", "μg/ml", "mg/l", "ppb", "ppm", "mol/l" etc. You can also enter the unit by yourself in the case of no proper unit existing in the concentration unit list.

"**Sample identification (ID)"** and "**Serial number"** generally make up the sample number. For example, Sample no. "S1" stands for the sample which has a **Sample ID** "S" and a **Serial number** "1".

After you finish the standard parameter settings, click "**Next step**" entering the page for **standard concentration setup** referring to the following figure. You can enter the concentration values into the concentration column of the standard list. Click "**Increase**" to add the standard samples, you may add up to 8 samples in the standard list. Click "**Decrease**" to remove standard samples, you should leave at least one standard sample in the list.

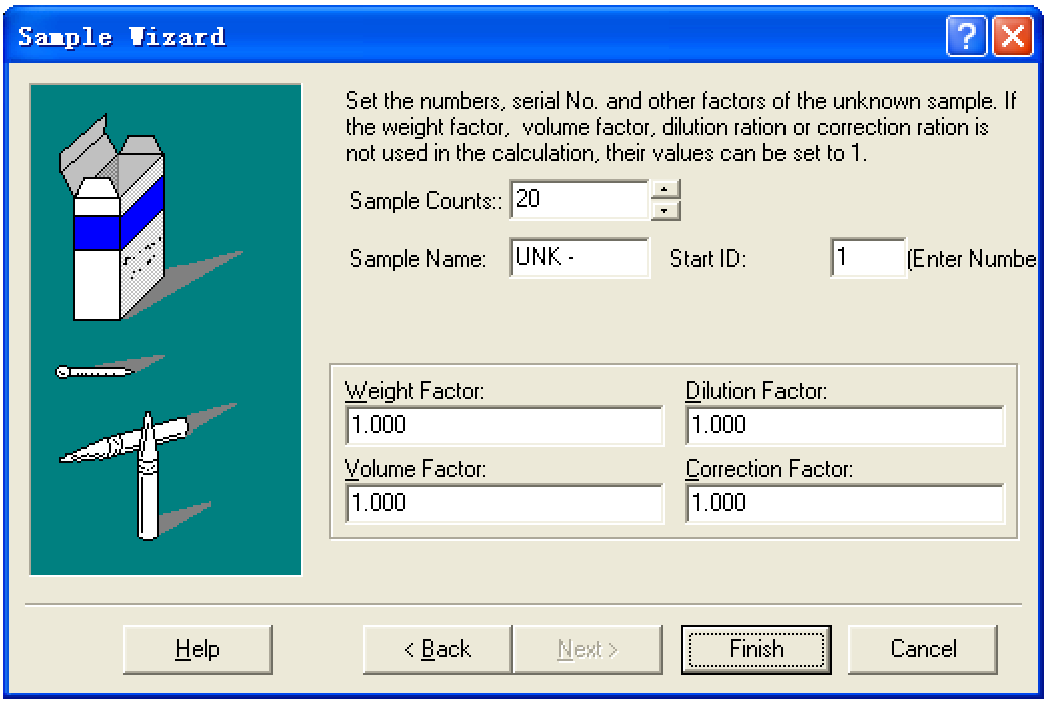


Sample Wizard

Set the number of the samples by using "Increase" or "Decrease" button. Edit or input the concentration of the sample in the table.

No.	Conc []
1	0.000
2	1.000
3	2.000
4	3.000

After finishing the standard concentration setup, click "**Next step**" to enter into the last page of the sample setup guide— the "**unknown sample setup**" page, see figure as follows:



Sample Wizard

Set the numbers, serial No. and other factors of the unknown sample. If the weight factor, volume factor, dilution ration or correction ration is not used in the calculation, their values can be set to 1.

Sample Counts:: 20

Sample Name: UNK -

Start ID: 1 (Enter Numbe

Weight Factor: 1.000

Dilution Factor: 1.000

Volume Factor: 1.000

Correction Factor: 1.000

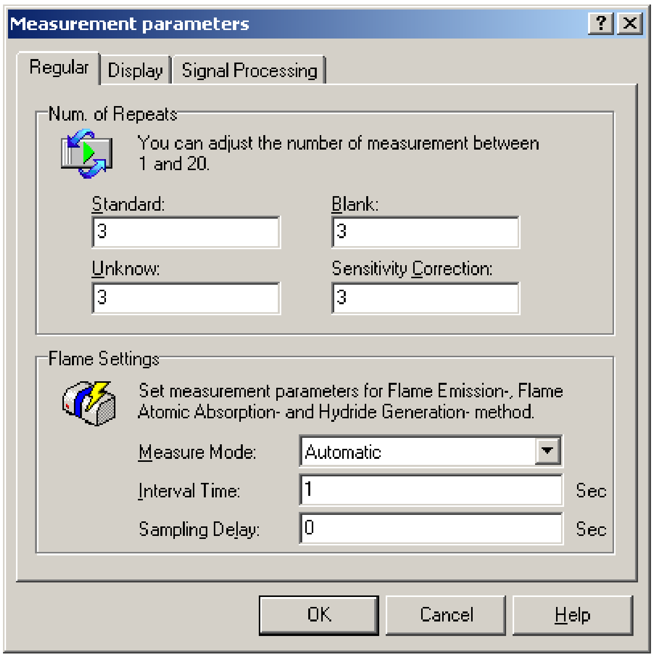
In this page, the following items will be set: number of unknown samples, sample ID and a variety of coefficients or factors needed for concentration calculation or correction. In the measure result table, one column entitled "**Actual concentration**" is listed. Its calculation formula is as follows:

$$\text{Real Concentration}=\frac{\text{Conc*Volume Factor*Dilution Ratio*Correction Factor* Sensitivity Correction coefficient}}{\text{Weight Factor}}$$

You can do the calculation of the actual concentrations through the proper selection of individual factors according to the practical sample situation. When you first open the sample setup guide, all the factors or coefficients are set to 1.000 as the default values, i.e. if you do not make any change, the actual concentration will be the same as that calculated from the absorbance of the sample in the end. If you need to modify your settings, click the “**former step**” button to edit the contents of the former pages. When you confirm that every sample parameter has been properly set and click the “**Finish**” button, system will transfer your settings into a measure table. You may next start to make a measurement.

Step 6: Set the measure parameters

Before making a measurement, you need to set the measure parameters first. Select [**Settings**] — [**Measurement parameters**] from the menu or click the button from the toolbar, and you will open a dialog box for measure parameter settings. The dialog box contain totally three optional pages, the first page is used to make regular parameter settings. See Figure as follows:



You can adjust the number of measurement between 1 and 20.

Standard: 3

Blank: 3

Unknow: 3

Sensitivity Correction: 3

Set measurement parameters for Flame Emission-, Flame Atomic Absorption- and Hydride Generation- method.

Measure Mode: Automatic

Interval Time: 1 Sec

Sampling Delay: 0 Sec

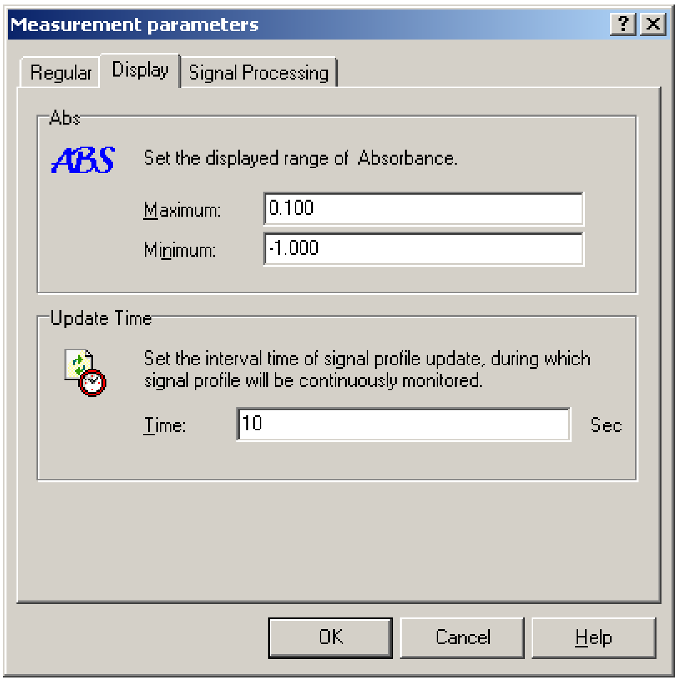
You may set duplicate numbers for the “**Standard**” and “**Unknown**” samples in the “**Repeat number for measurement**” section, the value of which is usually 1~3. If you set measure method to Flame Analysis (Flame absorption, Flame emission, Hydride generation method), you can select “**Manual**” or “**Automatic**” as the “**Operation mode”.** In the “**Manual**” mode, you need to press the keyboard manually to perform every individual measurement. In the “**Automatic**” mode, however, system will automatically finish all the repetitive measurements through pressing the keyboard only once at the very beginning. If you have chosen the “**Automatic**” mode, you need to set the “**Time interval**” and ”**Sampling delayed**” parameters.

“**Time interval**” is the waiting time for each sample uptake, it is usually set to be of 1~3 seconds.

“**Sampling delayed**” is the waiting time taken before auto-sampling, i.e. the pre-spraying time. It is usually set to be of 0 to 5 seconds.

After you finish the regular parameters, you may change to the second optional page

“**Display setup**” to make the display setup for measure graphs. Refer to the following figure.



Measurement parameters

Abs

Set the displayed range of Absorbance.

Maximum: 0.100

Minimum: -1.000

Update Time

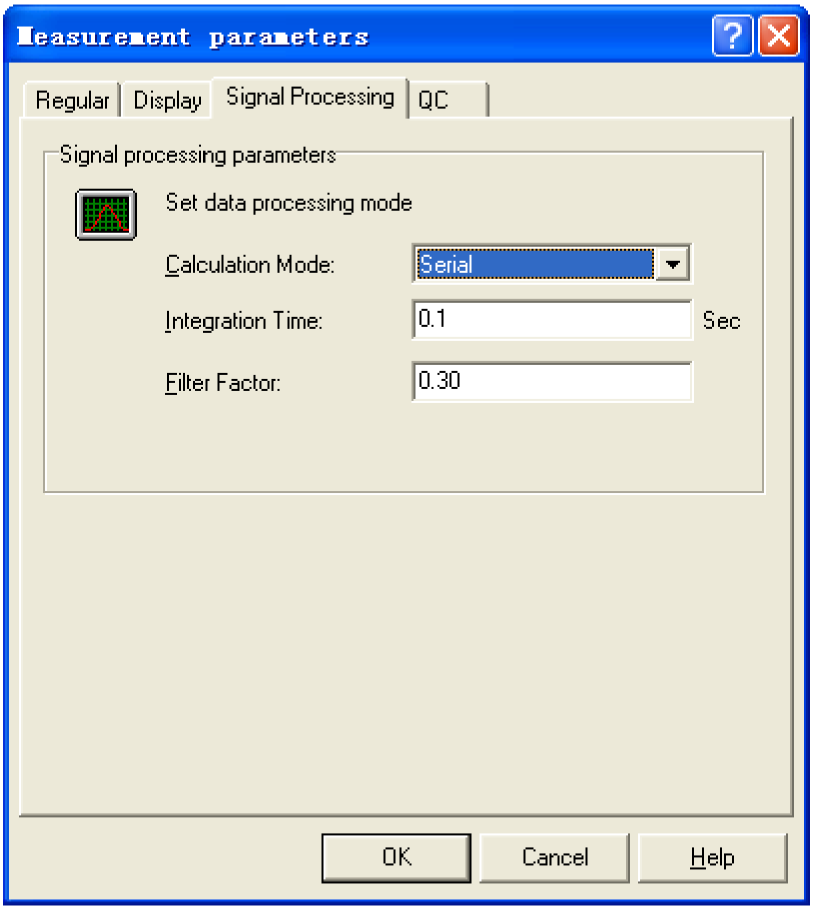
Set the interval time of signal profile update, during which signal profile will be continuously monitored.

Time: 10 Sec

In this page, you can set the display boundary of absorbance in the measure graphs, i.e. the lower and upper limits of the vertical / longitudinal axis. Generally, a lower limit with – 0.1 and an upper limit with 0.6 are set.

“Refresh time” is the maximum time period during which the real time profile can continuously be displayed in the measure graph in flame mode, namely the threshold of the time axis. It is generally set to be 300 seconds, that is, the real time profile will be renewed every 300 seconds,

The third page “**Data processing**” is used to set the signal processing parameters, refer to the following figure:



Calculation Mode: Serial

Integration Time: 0.1 Sec

Filter Factor: 0.30

In this page, you can choose the data calculation mode from the “**Calculation mode**” pull down menu box. In flame mode, the calculation mode “**continuous”** is commonly used**;** in furnace mode, however, “**peak height**” or “**peak area**” is often selected.

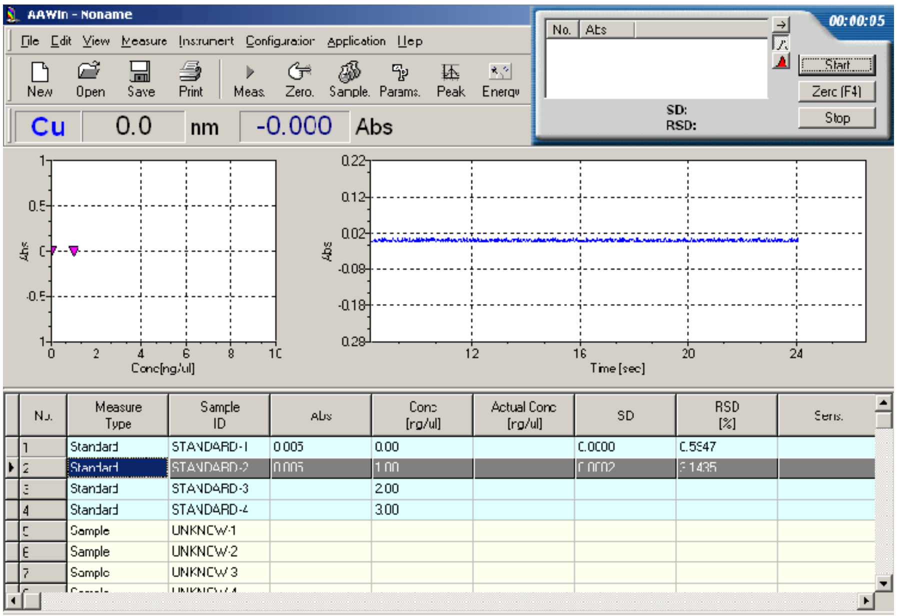
Input the integration time for your measure data in the “**Integration time**” box. In flame mode, an integration time of 1-3 seconds is usually selected. However, you need not to set the integration time in furnace mode. In that case, system will automatically take the total sum of the heating time in the atomization stage and the last three seconds in the former step (the ashing stage) from the furnace temperature program as the default integration time.

“Filter factor” is a constant which needs to be given to filter the acquired data, i.e. the flatten factor. In flame mode, a filter factor of 0.1~0.6 is often used, in furnace mode, it is usually set to be 0.1.

Step 7: Perform Measurement

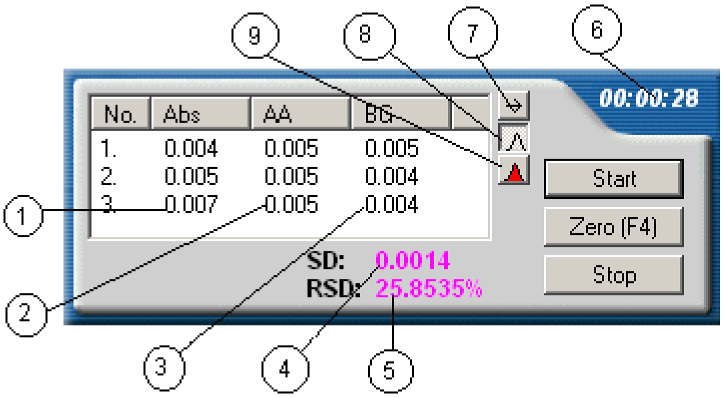
A． Flame analysis

In the case of flame analysis, before you make measurement, switch on the power of the air compressor and regulate its air exit pressure to 0.2-0.25Mpa, turn on the acetylene gas cylinder and carefully check all the piping tubes and the water seal for gas leak. When you make sure that everything is O.K, select [**Application**]—[**Ignition**] from the menu or click the “⚡” button from the toolbar for igniting the flame. When the flame height is too high or too low or if the burner is not set in proper position, turn to the “**Burner parameter setup**” for adjustment. In the next, select [**Measurement**]—[**Start**] from the menu or click the button “▶” from the toolbar or just press F5 key to open the measurement window, then there will be analog signal coming up in the real time graph. Refer to Figure as follows:



No.	Measure Type	Sample ID	Abs	Conc [ng/ul]	Actual Conc [ng/ul]	SD	RSD [%]	Sens.
1	Standard	STANDARD-1	0.005	0.00		0.0000	0.5947	
2	Standard	STANDARD-2	0.005	1.00		0.0002	3.1435	
3	Standard	STANDARD-3		2.00				
4	Standard	STANDARD-4		3.00				
5	Sample	UNKNOW-1						
6	Sample	UNKNOW-2						
7	Sample	UNKNOW-3						

Put the blanks, the standard solution series and the unknown samples as well in the front of instrument, insert the sampling capillary into the measure sample solution until the displaying data or signal is stable, then click the [**Start**] button for sampling. If you have chosen a **Manual** mode from the measure parameter setup window, you need to press the [**Start**] button again for another measurement after you finish every single sampling operation. In the case of **Automatic** mode, however, system will automatically take the sampling process according to your setting of the "**Time interval**". After you finish the sampling, system will display the results in the measure window, and standard deviation (SD) and relative standard deviation (RSD) values will also be calculated and displayed in the window after the third repetitive sampling. After you perform the sampling for the set number of measurement, system will calculate the mean value of the measure results and display it in the proper cell of the measurement table. See also the following figure:



No.	Abs	AA	BG
1.	0.004	0.005	0.005
2.	0.005	0.005	0.004
3.	0.007	0.005	0.004

SD: 0.0014
RSD: 25.8535%

① Absorbance(Net value)

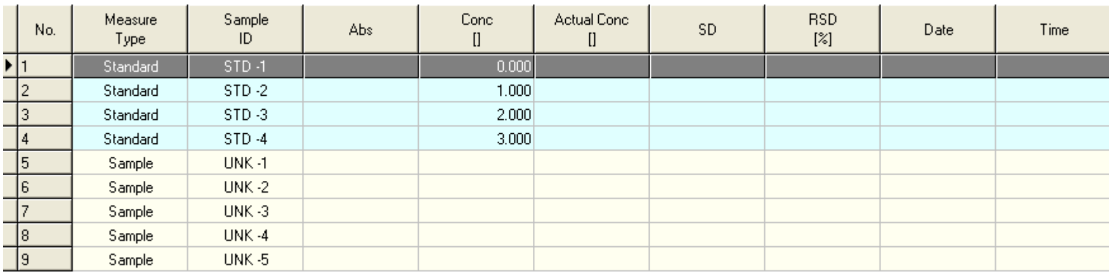
② Atomic absorption value(AA value)

③ Background value(BG value)

④ Standard deviation

⑤ Relative standard deviation

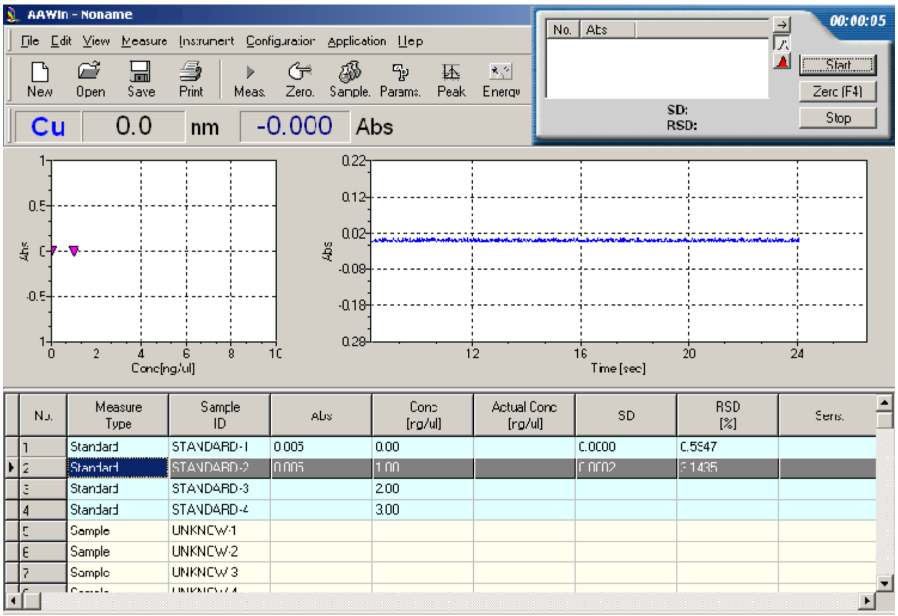
⑥ Measurement timer
⑦ Results for continuous integration mode
⑧ Results for peak height calculation mode
⑨ Results for peak area calculation mode



No.	Measure Type	Sample ID	Abs	Conc []	Actual Conc []	SD	RSD [%]	Date	Time
1	Standard	STD -1		0.000					
2	Standard	STD -2		1.000					
3	Standard	STD -3		2.000					
4	Standard	STD -4		3.000					
5	Sample	UNK -1							
6	Sample	UNK -2							
7	Sample	UNK -3							
8	Sample	UNK -4							
9	Sample	UNK -5							

During measurement, you can check the total time used for your analysis through a timer placed on the upper right of the measurement window. You can also insert a blank before each sampling for zero check. Click the “**Zeroing**” button to make zero adjustment. Press the “**End**” button when finished the measurement. If you have selected D_2 lamp or SR mode for the background correction, AA and BG values will be displayed together with the absorbance (net value) in the measurement window. In the mean time, the AA and BG curves will also appear in the real time graph. In this case, the absorbance is the difference subtracting the BG value from the AA value (Abs. = AA — BG)。If no background correction is used, system will display the absorbance and provide the Abs. Curve only. In this situation, the abs. is equal to the AA value (Abs. = AA). Curve color in the real time graph can be set through the function of the “**Graph Setup**”. In the case of default, Abs. Curve takes blue, AA curve is light green and BG curve for red.

When measuring the standard samples, system will draw up each point of the standards on the graph of the calibration curve after sampling, and will display the final calibration curve on the graph when all the standards are finished. See Figure as follows:

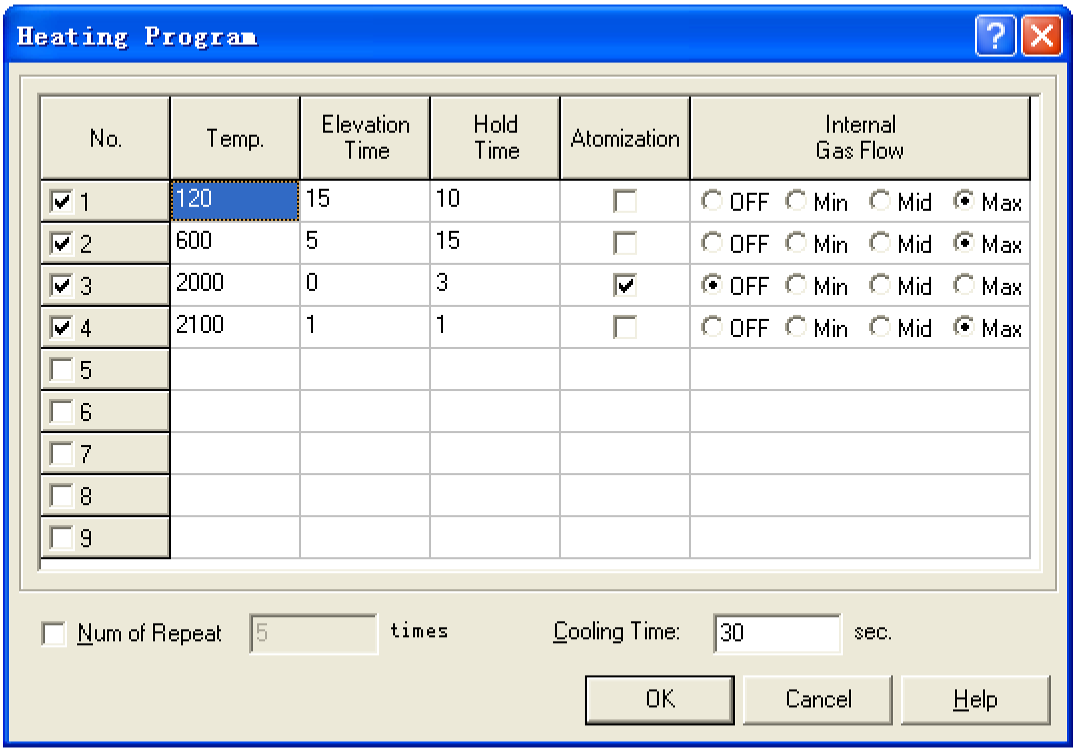


No.	Measure Type	Sample ID	Abs	Conc [ng/ul]	Actual Conc [ng/ul]	SD	RSD [%]	Sens.
1	Standard	STANDARD-1	0.005	0.00		0.0000	0.5947	
2	Standard	STANDARD-2	0.005	1.00		0.0002	3.1435	
3	Standard	STANDARD-3		2.00				
4	Standard	STANDARD-4		3.00				
5	Sample	UNKNOW-1						
6	Sample	UNKNOW-2						
7	Sample	UNKNOW-3						

After you finish the measurement of your standard sample series, double click the "**calibration curve"** box to check your calibration curve. If there is any point showing a large offset from your calibration curve, you can measure it for a second time. After preparing the calibration curve, you may next perform the measurement of your unknown samples. The measurement results will also be auto-filled into the individual unit elements of the measurement table. In AAWin software system, measurements are made sequentially according to the measurement table. After you finish your current sample, click "**Start**" from the measurement window for the next. The current measuring sample in the measurement table will be automatically changed to the next. If you are measuring the last sample as for the current sample, system will add a new unknown sample in the measurement table for you to continue your measurement.

B. Graphite furnace analysis

Before measurement, please carefully check all the gas and water piping tubes for leak. When you are complete, switch on the cooling water supply and open the argon gas cylinder with its exit pressure set on 0.5MPa. For furnace analysis, you need to set the heat program first. Select [**Setup**]—[**Set furnace heat program**] from the menu or click the button [**Heat**] to open the "**furnace heat program setup**" box, see the following figure:



No.	Temp.	Elevation Time	Hold Time	Atomization	Internal Gas Flow
☑ 1	120	15	10	☐	○ OFF ○ Min ○ Mid ● Max
☑ 2	600	5	15	☐	○ OFF ○ Min ○ Mid ● Max
☑ 3	2000	0	3	☑	● OFF ○ Min ○ Mid ○ Max
☑ 4	2100	1	1	☐	○ OFF ○ Min ○ Mid ● Max
☐ 5					
☐ 6					
☐ 7					
☐ 8					
☐ 9					

☐ Num of Repeat 5 times

Cooling Time: 30 sec.

The furnace heat program generally includes four temperature elevation stages namely drying, ashing, atomizing and clearing.

Drying: temperature 100-120℃， heating time 5-20s，hold time 5-20s，inner gas flow rate is set to high；

Ashing: temperature 200-2000℃ (according to the measure element)，heating time 1-15s，hold time 5-15s，inner gas flow rate is set to high；

Atomizing: temperature 1400-2700℃ (according to the measure element)，heating time 0-1s，hold time 2-5s，inner gas flow rate is set to close/none；

Clearing: temperature is equal to or slightly higher than the atomization temperature，heating time 0-1s，hold time 1-2s，inner gas flow rate is set to high.

Note: After setup of the heat program, you must click “confirm” to exit.

When you finish the temperature program, first select [**empty burn**] to burn for at least twice from toolbar. then select [**Measurement**]—[**Start**] from the menu or click “▶” from the toolbar or just press F5 key. System will change to the measurement window.

For furnace analysis, no signal profile will be displayed in the measurement window before you click the [**Start**] button. Using a pipette load your sample of 10-20μl into the graphite tube, then click [**Start**] for running the furnace temperature program. In this time, signals begin to appear in the measurement window, and the current furnace temperature, heat stage together with a timer, which will indicate the time left for the heat stage, will also be displayed in the window. Refer to Figure as follows:

No.	Abs	AA	BG
1.	0.002	0.000	0.003
2.	0.011	0.005	0.002
3.	0.009	0.005	0.003

Step 4 of 4
(0) 0 °C
SD: 0.0047
RSD: 63.6404%

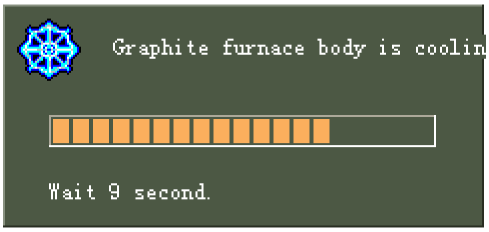
①The current heat step

②Number of the total heat steps

③Timer for how much time is left for the current heat step

④The current temperature

After each measurement, a countdown clock for furnace body cooling down will pop up. During this period, no other operations can be performed through the software. You have to wait until the timer is over before performing the next measurement. See figure as below.



Graphite furnace body is coolin

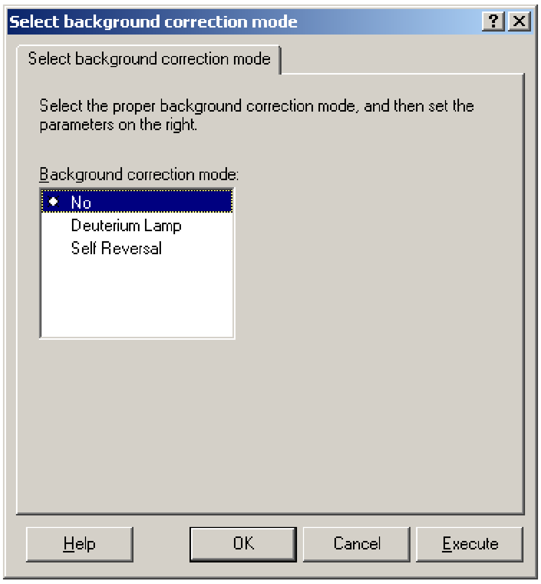
Wait 9 second.

When modifying the furnace temperature program or the other instrumental parameters, first end up the current instrumental operation and close the measurement window. After the modification is complete, you can re-enter into the measurement window to continue your measuring operation. The data processing and display for the measurement results in furnace mode are the same as in flame mode.

Step 8: Select the background correction mode

A. D_2 lamp background correction

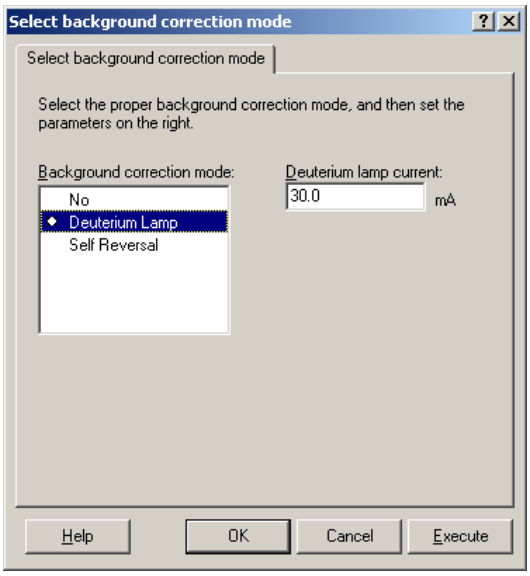
In the measurement of atomic absorption analysis, if the measured sample contains too much base element, causing a serious problem of spectral interference, in this case, you need to use the background correction mode for measurement. The background correction modes that are available for AA-990 series atomic absorption spectrophotometer, include the D_2 lamp-BGC and SR lamp-BGC mode. You can select [**Instrument**] — [**Background correction mode**] from the menu to open the background correction mode selection box, refer to the following figure.



Select the proper background correction mode, and then set the parameters on the right.

Background correction mode:
- No
- Deuterium Lamp
- Self Reversal

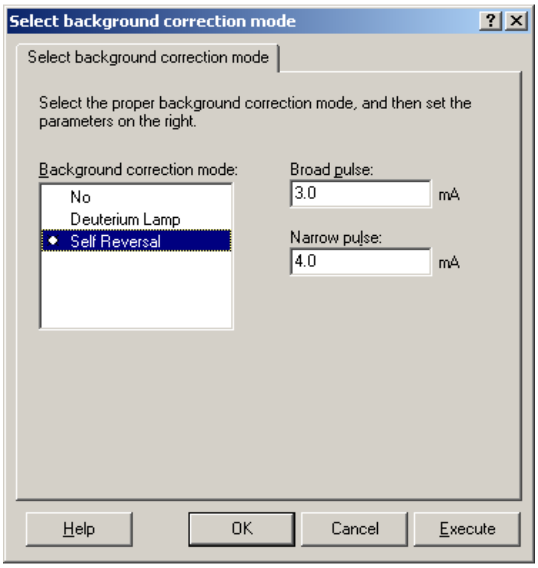
You can choose the D_2 lamp background correction mode from the list box and click [**Determine**] or [**Execute**], system will immediately set the D_2 lamp mode as the current background correction mode. From its current input box you may set the D_2 lamp current according to your own requirements. The D_2 lamp current is usually set to a value of 20-60mA. When you choose SR lamp to perform the background correction, you can enter the wide and narrow pulse current values for the SR lamp in their input boxes. Refer to Figure as follows:



Select the proper background correction mode, and then set the parameters on the right.

Background correction mode: ◆ Deuterium Lamp

Deuterium lamp current: 30.0 mA



Select the proper background correction mode, and then set the parameters on the right.

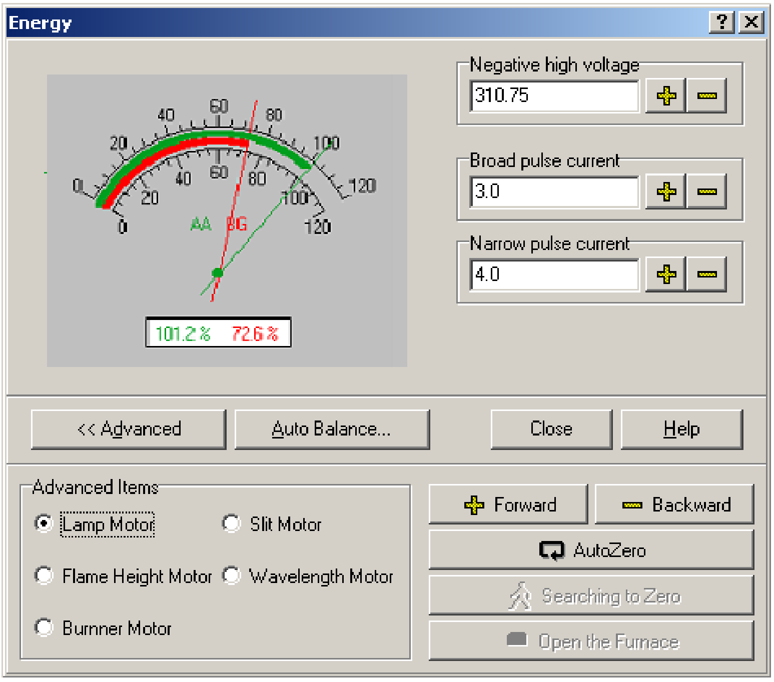
Background correction mode: ◆ Self Reversal

Broad pulse: 3.0 mA

Narrow pulse: 4.0 mA

Note: When D_2 lamp is used to perform the background correction, it should be always avoided that D_2 lamp is lit up under a large current for long time. When finish your measurement, shut down the lamp power as soon as possible in order to extend its useful life period.

In the case of D_2 lamp as the background correction mode, you need to perform the beam balance, adjusting the D_2 lamp energy to be matched with that of the element HCL lamp. From the system shortcut icons, click the "**Energy**" icon to open the energy adjustment box. Click "**Advanced adjustment**" to pop up the advanced adjustment box, referring to the following figure. Select the D_2 lamp mirror motor and click the "**positive rotation**" or "**Reverse/Back turn**" button to adjust the energy of the D_2 lamp to its maximum position. Press. When finished, check the adjustment result. If the D_2 lamp current is larger than 120mA, reduce the element lamp current and click "**Auto energy balance**" again for a second beam balance in order to extend the D_2 lamp's service life.



101.2 % 72.6 %

Negative high voltage: 310.75

Broad pulse current: 3.0

Narrow pulse current: 4.0

Advanced Items

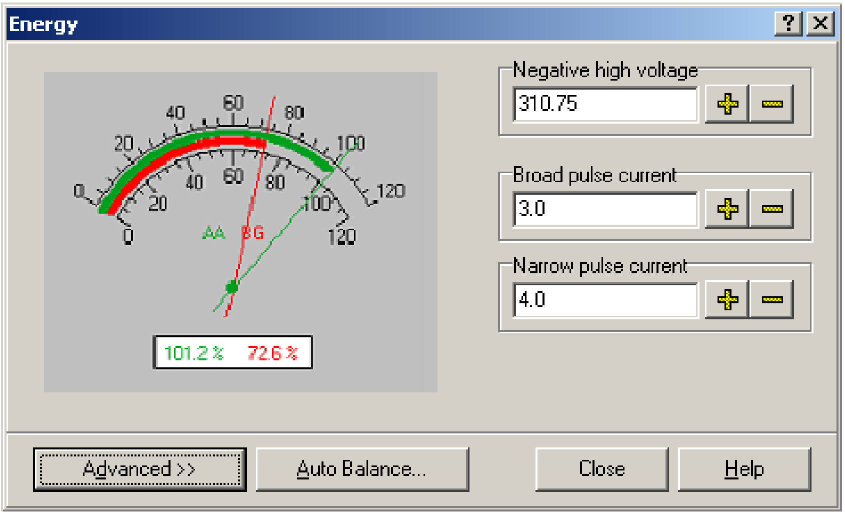
● Lamp Motor ○ Slit Motor

○ Flame Height Motor ○ Wavelength Motor

○ Burnner Motor

B. SR background correction

When you choose SR background mode, click “Auto energy balance” to perform the beam balance. See the following figure.



101.2 % 72.6 %

Negative high voltage: 310.75

Broad pulse current: 3.0

Narrow pulse current: 4.0

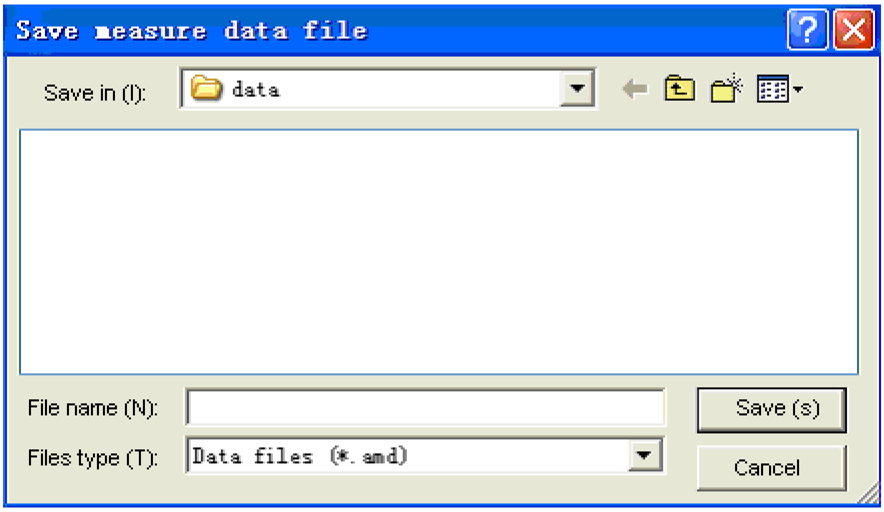
You also can input the lamp current and high voltage to perform energy balance.

When you complete performing background correction, switch the current background correction mode to the none BGC mode as soon as possible to reduce the lamp deterioration.

5.3 Data processing and report output

After measurement, you may perform data processing for the individual measured results and make a report output for the measured results. Contents of each parameter setting and measurement results can be output to the printer and stored.

If you need to save your measured results in a file, select **[File]—[Save]** from the menu or click the “💾” button from the toolbar, see figure as follows.



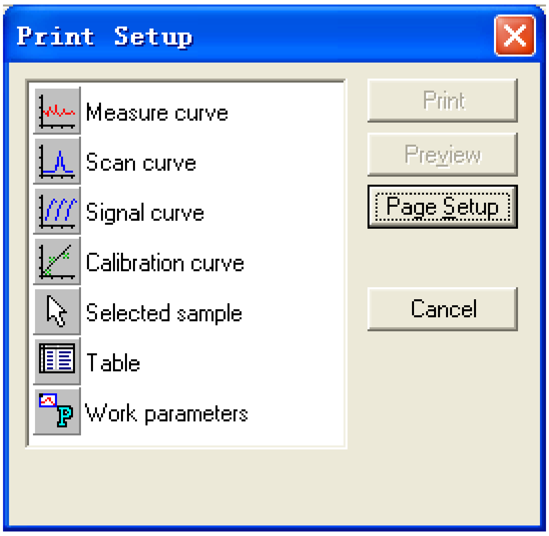
Save in (I): data

File name (N):

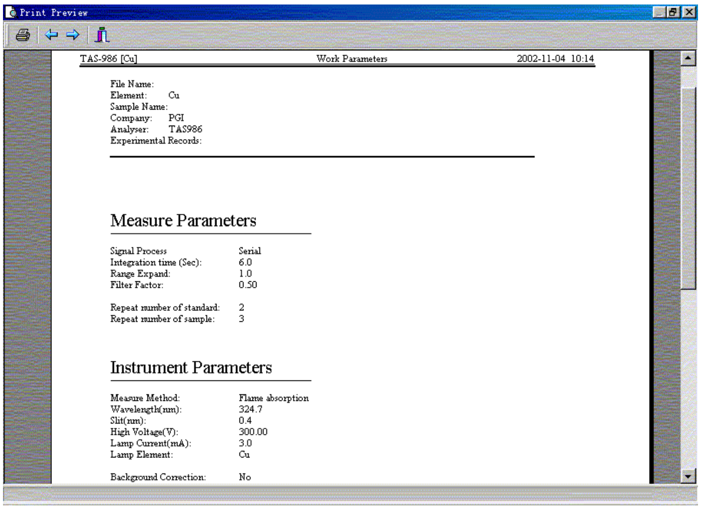
Files type (T): Data files (*.amd)

“**Save**”: Store the content of the current measured results table in a file. When selected the “**Save**” function, system will pop up the “**Save as a file**” dialog box, refer to figure above. Enter a file name to be stored in the “**File name**” box and click “**Save**”, system will store the measured results in the specified file. If the file name you have input has already existed, system will prompt a warning message to ask you weather you want to overwrite it or not. When choose “**Yes**”, the existing file will be overwritten. Otherwise, system will cancel the operation of saving the measurement results.

“**Print**”: This function provides you with outputting your data and graphs to the printer. Selecting “**Print**”, system will pop up the ”**Select the output report type**” box, see figure as below. Specify the type of the output report and click “**Determine**”, the contents of the current output report will be printed out.



- **Measure curve**: This function allows outputting the current measure curve to the printer.
- **Signal curve**: Selecting this function, the current signal profile will be printed out.
- **Calibration curve**: The current calibration curve will be printed out.
- **Search peak/Scan curve**: This function is to print out the current wavelength scan or line search graph.
- **Selected sample**: The sample data in the current cursor position of the measured results table will be printed out.
- **Measure table**: This function will allow all data in the measured result table to be printed out.
- **Work parameters**: This will allow you to output the current operation conditions of the instrument together with the measure parameters to the printer. If you need to preview the contents to be printed out, click “**Preview**” to open the “**Print preview**” window, as shown in the following figure.



TAS-986 [Cu] Work Parameters 2002-11-04 10:14

File Name:
Element: Cu
Sample Name:
Company: PGI
Analyser: TAS986
Experimental Records:

Measure Parameters

Signal Process Serial
Integration time (Sec): 6.0
Range Expand: 1.0
Filter Factor: 0.50

Repeat number of standard: 2
Repeat number of sample: 3

Instrument Parameters

Measure Method: Flame absorption
Wavelength(nm): 324.7
Slit(nm): 0.4
High Voltage(V): 300.00
Lamp Current(mA): 3.0
Lamp Element: Cu

Background Correction: No

When you need to print them out directly, click the “🖨” button. In the case that a measured table is selected, you can use the “⇦” or “⇨” button to browse through the entire measurement data in the table. Click “” to close the “**Print preview**” window.

5.4 End the measurement

When finish the flame analysis, turn off the acetylene gas supply and spray the blank solution for several minutes to clean the nebulizer, and then switch off the air compressor. When finish the graphite furnace analysis, turn off the argon gas supply and switch off the cooling water. Check and clean out all the standard and unknown samples and organize your measured results in the report.

5.5 Exiting the AAWin system

When you have finished all the measurements and made a print-out of the measure results or have saved them to the disk, you can select [**File**]—[**Exit**] from the menu to quit from the AAWin software system. Before closing AAWin system, a message box will pop up to remind you to follow the proper procedure to shut down the instrumental system. This warning message will always come up every time you close the AAWin software system in case that you may forget to shut down the water, gas or electricity supply. Therefore, you are supposed to follow exactly the shut-down procedure to quit from the analysis, i.e. after finishing measurement and closing the AAWin software, switch off the water, gas and power supply immediately in case of any accident.

Chapter 6 Maintenance and trouble-shooting

6.1 Diagnose and maintain of some troubles

For the instrument is a precise instrument, the user is not allowed to disassemble or modify the equipment. But the user can solve some simple troubles caused by improper use.

Please contact with the manufacturer if you met troubles which you can’t solve by yourselves.

6.2 Common troubles and their solutions

A. The instrument is not electrified

Trouble phenomenon: the power supply makes no response.

Check step by step:

5. Check whether the plug of the power supply is dropped off or loosen.
6. Check the power-supplied equipment such as the plug of the power is electrified, and the connection of the plug is OK.
7. Check whether the power supply is qualified.
8. Check whether the 3A fuse is blown out.
9. Check whether the power supply wiring is damaged, if it is, change another one.

B. The wavelength motor is failed when initialization

Trouble phenomenon: the sign “✕” appears when initialization.

Check step by step:

1. Check whether the element lamps is normally installed and lit.
2. Check whether the optical path is blocked.
3. The communication between the main unit and the computer is interrupted abruptly. Check it by restarting.

C. The element lamp is not lit or working improperly

Trouble-shooting methods

1. Check if the lamp connection plug is loose or off.
2. Check if the power connection is loose or off.
3. Check if the element lamp is damaged.

D. The energy is low for peak searching and the high voltage is overflow

Trouble-shooting methods

1. The element lamp is not lit.
2. The facula of the element lamp is not entered into the optical path, or the element lamp is not at the optimum position.
3. The peak-searching wavelength selected is not the characteristic spectral line of the element.
4. Check if there is any obstacle in the optical path.

5. The element lamp is ageing so that the energy is too low.
6. The element lamp parameter “wavelength” is not selected correctly.
7. Restart the main unit and test again.

E. Abnormal ignition and extinguish

Trouble-shooting methods

A. Trouble phenomenon: The igniter does not discharge by pressing the IGNIT button.

Check step by step:

1. Check if the power for the air compressor has been switched on and if the exit air pressure is too low (lower than 0.2Mpa)；
2. Check if there is any intensive light irradiating on the flame burner head; and check if the detector of the flame status is work normally;
3. Check whether the burner head is installed correctly.
4. Check whether the waste fluid equipment is filled with fluid.
5. Check whether the switch of the emergency extinguishing is displayed.
6. Check if it alarms when acetylene leaks.

B. Trouble phenomenon: the flame is not lit when click “ignition” and the igniter does discharge.

Check step by step:

1. Check whether there is flame spurted out, if there is, it can be judged that there is no acetylene gas in the pipeline of the instrument. Check if the acetylene gas cylinder is open and the pressure is appropriate; check whether the acetylene gas pipeline is too long; you can check whether the acetylene gas entered into the instrument by ignition time after time.
2. Check whether there is any extensive light irradiating on the flame burner head:
    a) Check whether the burner is at the appropriate position.
    b) Check whether the gas flow is more than 1500ml/min，open “burner parameters setting” to confirm.
    c) Check whether the assistant flame spray distance is proper.
    d) Check whether the exit pressure of the air compressor is proper.

F. Low energy for background or no energy when selecting deuterium background

Trouble-shooting methods

1. Check if the deuterium lamp is lit; observe the facula of the deuterium lamp；
2. Check whether the wavelength of the instrument is set under 320nm.
3. Check if the half-transmitting-half-reflecting mirror for the deuterium lamp rotates to the proper angle so that the deuterium facula and the element lamp facula overlap. Observe it with a piece of white paper blocking in the optical circuit. Adjust the counter rotation and contrarotation by single step of the deuterium lamp motor to make two faculas overlap. If the faculas don’t overlap, then open the left cover of the instrument and adjust the setting screw to make the faculas overlap.

G. Unstable baseline and too much noise is detected

Trouble-shooting methods

1. Check if the lamp energy is too low or if there is a much high output voltage for the photomultiplier or a very large current value for the H.C. Lamp；
2. Check if there exists a large fluctuation for the output voltage of the AC 220V power, which

causes too much noise；

3. Check if the hollow cathode lamp for the element is defective. Replace a new one and check it again；
4. Check if the DC power for the instrument gives too much noise or if the voltage monostat/regulator is defective；
5. Check if there is any strong vibration source around the instrument；
6. Check if there is any high power consumption device running nearby the laboratory, which exerts a large impact on the power network causing the baseline unstable；
7. Check if the instrumental circuit board is defective.

H. Low Abs value or no Abs value

Trouble-shooting methods

1. Check if the optical axis has been properly adjusted and if the optical path is running through the center of the burner slit；
2. Check if the flame height has been properly set；
3. Check if the fuel/combustion to support gas ratio is appropriate;
4. Check if the wavelength has been properly set on the position of the characteristic spectral line for the specified element;
5. Check if the energy level for the element lamp is too low or overflowed;
6. Check if the pipettor is clogged and if the amount of sample uptake or the spray rate is proper. Replace the nebulizer to test the atomization efficiency or the spray rate;
7. Check if the nebulizer is defective or clogged；
8. Check if the concentration of the element in solution is too low；
9. Check if the flame is stable and if other conditions are appropriate；

I. Flame unstable during test

Trouble-shooting methods

1. Check if the exit pressure of the air compressor is stable or not;
2. Check if the acetylene flow is stable or not;
3. Check if the pressure of the acetylene cylinder is too low;
4. Check if there is any salt substance precipated on the burner and blocked the burner slit;
5. Check if there is anything clogged in the drainage tubing system;
6. Check if the water sealing in the waste tubing system is defective;
7. Check if the exhaust rate for the ventilation system is set too large;
8. Check if there is any wind blowing around the instrument.

J. BGC compensation too low or insufficient in case of the D2 lamp background correction mode

Trouble-shooting methods

1. Mainly check the two beams from the element lamp and the deuterium lamp are completely overlapped.

K. The lamp energy made no response to the change of high voltage of the photomultipliefier

Trouble-shooting methods

1. Check if the energy of the instrument is saturated or overflowed the upper limit;
2. Check if the AA host instrument is disconnected with the computer or if the communication between them is interrupted, which result in the computer being in off-line;

L. AA host instrument made no response to the computer software program

Trouble-shooting methods

1. Check if the AA host instrument has been disconnected with the computer or if the communication between them is interrupted, which makes the computer being in off-line;
2. Check if the AA host instrument is busy responding to other operational instructions and has not yet ended;
3. Check if the communication cable is loose;
4. Check if the computer is down；

M. The energy output is too low or not detected in case of measurement

Trouble-shooting methods：

1. Check if there is anything blocking in the beam path;
2. Check if the high voltage power is in proper state;
3. Check if the computer is in dead lock or disconnected in off-line to the AA host instrument because of communication interruption

N. The wavelength is shifted or its reading accuracy does not satisfy the specification of no more than ±0.3nm

Trouble-shooting method：

1. Use the “wavelength correction” button for the AAWin software system to make adjustment. Details about the operation of wavelength correction please refer to section 5.5 wavelength correction in this instruction manual.
2. Please contact with the supplier if the deviation is still more than ±0.3nm.

6.3 Problems concerning the furnace heat control

A. Furnace body cannot be automatically opened and closed during graphite tube exchange

Trouble-shooting method：

1. Check if the exit pressure of argon cylinder is properly regulated in 0.4~0.5Mpa and if argon supply is sufficient；
2. Check if there is anything blocking in the gas flow path or if there is any significant bending and folding in the gas flow hose;
3. Check if the power connections between the host control part of the instrument and the furnace are properly attached；
4. Check if the instrument is off-line.

B. Abnormal heat control status for the furnace heat program

Trouble-shooting method:

1. Check if the instrument is off-line.
2. Check if the cooling water flow is more than 1L/Min or if the argon pressure is higher than 0.5 Mpa.
3. Check if the power connections between the host control part of the instrument and the furnace are properly attached；
4. Check if the pulse signal output for the main PC circuit board is normal；
5. Check if the furnace power has been switched on and if the control signals are proper；
6. Check if the power supply of the graphite furnace is on.
7. Check if the safety switch for the furnace power supply has been turned on and if the fuse is blown out.

C. Absorbance is too low or absent during the GF-AAS analysis

Trouble-shooting method：

1. Check if the facula for the element lamp is on the optical axis passing through the center of the graphite tube；
2. Check if wavelength has been properly set on the position of the characteristic spectral line for the specified element；
3. Check if the lamp energy is too low or actually already saturated;
4. Check if the elevation and hold time for the individual stages of the furnace heat program such as drying, ashing, atomizing and cleaning-up etc, have been properly set；
5. Check if atomization has been properly set in the furnace heat program;
6. Check if the main inner gas flow and/or the supporting gas flow have been switched off during the atomization process;
7. Check if the time for integration is well matched for the atomization process;
8. Check if the graphite tube is defective or becomes badly aged being oxidized and damaged when used too often.

D. Furnace body is extremely hot during the GF –AAS analysis

Trouble-shooting method：

1. Check if the cooling water flow is more than 1 L/min；
2. Check if the water is flowing smoothly through the cooling tubing system and if there is anything blocked in the water flow path of the water-cooled electrode;
3. Check if the temperature and the heating time for the atomization and /or its following clean-up stage in the furnace program have been properly set. Make sure that the total time for heating under the temperature higher than 2500℃ is no more than 10s.

E. Gas flow path blocked, no gas can flow through or be cut off during the furnace test

Trouble-shooting method：

1. Check if the power control connections from the furnace has been properly attached and if there is any pin connection bending or broken.
2. Check if there is any solenoid valve (electromagnetic valve) defective and if the regulator with which the solenoid valve is attached, is extremely offset or the connections for the solenoid valve are improper. Unpack the main unit cover of the graphite furnace for check and adjustment.